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INVESTIGATION OF TANTALUM AND ITS ALLOYS

TECHNICAL DOCUMENTARY REPORT NO. ASD-TDR-62-594, PART II
May 1963

AF Materials Laboratory
Aeronautical Systems Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

Project No. 7351, Task No. 735101

(Prepared under Contract No. AF 33(657)-7927
by Battelle Memorial Institute, Columbus, Ohio;
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FOREWORD

This report was prepared by Battelle Memorial Institute under USAF Contract No. AF 33(657)-7927. This contract was initiated under Project No. 7351, "Metallic Materials", Task No. 735101, "Refractory Metals". This project was administered under the direction of the Air Force Materials Laboratory, Deputy Commander/Research and Engineering, Aeronautical Systems Division, with Lt. William E. Smith and Mr. James T. Gow serving as project engineers.

This report describes the results of research conducted during the period April 1, 1962, through February 28, 1963.

ABSTRACT

Minor (1 to 2 per cent) reactive metal (Zr, Hf) additions to Ta-W-Mo alloys effect pronounced strengthening at 1925 C (3500 F) with minimal degradation of low-temperature alloy behavior. Additions of carbon to alloys containing reactive metals degrade both 1925 C (3500 F) strength and low-temperature behavior; however, "ZrC dispersions" exhibit pronounced strengthening at lower temperatures [1480 C (2700 F)]. Solution-process anneals for alloys containing "ZrC dispersion" greatly increase strength at 1480 C (2700 F), but seriously impair fabricability and ductility.

Molybdenum is a less effective strengthener than tungsten as an alloying addition to tantalum. Tungsten additions provide higher stress-rupture strengths with less degrading effects on low-temperature ductility than do equivalent atomic percentages of molybdenum. Welding increases the ductile-to-brittle transition temperature of Ta-W-Mo alloys by 300 to 500 C (540 to 900 F).

Rhenium and ruthenium additions showed little or no superiority to tungsten as solid-solution strengtheners, when both high- and low-temperature effects were considered.

This technical documentary report has been reviewed and is approved.



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TABLE OF CONTENTS

| | <u>Page</u> |
|---|-------------|
| INTRODUCTION | 1 |
| SUMMARY | 2 |
| Dispersion Effectiveness | 2 |
| Effects of Fabrication on a Dispersion-Containing Alloy | 3 |
| Detailed Behavior of Solid-Solution Alloys | 4 |
| Effects of Rhenium and Ruthenium Additions | 5 |
| GENERAL PROCEDURES | 6 |
| Materials | 6 |
| Alloy Preparation | 6 |
| Melting | 6 |
| Rolling | 6 |
| Heat Treatment | 8 |
| Specimen Preparation | 10 |
| Metallographic Techniques | 12 |
| Mechanical Testing | 12 |
| PROGRAM DESIGN AND ALLOY SELECTION | 14 |
| EXPERIMENTAL RESULTS | 17 |
| Dispersion Effectiveness | 17 |
| Fabrication | 18 |
| Evaluation | 23 |
| Bend Tests | 23 |
| Tensile Tests | 23 |
| Stress-Rupture Evaluation | 26 |
| Recrystallization Behavior | 32 |
| Welding | 32 |
| Effects of Fabrication on a Dispersion-Containing Alloy | 32 |
| Fabrication | 32 |
| Evaluation | 47 |
| Tensile Tests | 47 |
| Recrystallization Behavior | 47 |
| Thermal Exposure | 52 |
| Detailed Behavior of Solid-Solution Alloys | 52 |
| Fabrication | 52 |
| Evaluation | 54 |
| Transition Behavior | 56 |
| Stress-Rupture Behavior | 56 |
| Recrystallization Behavior | 71 |
| Welding and Thermal Exposure | 71 |
| Effects of Rhenium and Ruthenium Additions | 93 |
| Fabrication | 93 |
| Evaluation | 93 |

TABLE OF CONTENTS
(Continued)

| | <u>Page</u> |
|--------------------------------------|-------------|
| Bend Tests | 95 |
| Tensile Tests | 95 |
| Recrystallization Behavior | 95 |
| Welding | 95 |
| DISCUSSION | 101 |
| CONCLUSIONS | 107 |
| RECOMMENDATIONS | 108 |
| REFERENCES | 109 |
| APPENDIX I | |
| MELTING DATA | 111 |
| APPENDIX II | |
| WELDING STUDIES | 115 |
| APPENDIX III | |
| RECRYSTALLIZATION BEHAVIOR. | 117 |

LIST OF TABLES

| <u>TABLE</u> | <u>Page</u> |
|--|-------------|
| 1. Analyses of Electron-Beam-Melted Tantalum | 7 |
| 2. Alloy Compositions Selected for Evaluation | 15 |
| 3. Repeat Alloy Compositions Selected for Evaluation of Dispersion Effectiveness | 16 |
| 4. Fabrication Data for Dispersion-Effectiveness Alloys | 19 |
| 5. Bend Ductilities of Dispersion-Effectiveness Alloys at 25 and -195 C (75 and -320 F) | 24 |
| 6. Room-Temperature Tensile Properties of Tantalum-Base Alloys | 25 |
| 7. Tensile Properties of Dispersion-Effectiveness Alloys at 1480 to 1925 C (2700 to 3500 F) | 27 |
| 8. Stress-Rupture Properties of Tantalum, Ta-5W-2.5Mo, and Ta-5W-2.5Mo-0.5Zr-0.07C at 1480 and 1925 C (2700 and 3500 F) | 28 |
| 9. Rupture Strength of Tantalum, Ta-5W-2.5Mo, and Ta-5W-2.5Mo- 0.5Zr-0.07C Alloys at 1480 and 1925 C (2700 and 3500 F) | 31 |
| 10. Recrystallization Temperatures for Dispersion-Effectiveness Alloys | 33 |
| 11. Microstructures and Hardnesses of Dispersion-Effectiveness Alloys | 34 |
| 12. Results of Examination of Automatic TIG-Produced Welds for Dispersion-Effectiveness Alloys | 35 |
| 13. Automatic TIG Weld-Bend Ductilities of Dispersion- Effectiveness Alloys | 36 |
| 14. Fabrication Data for the Ta-5W-2.5Mo-0.5Zr-0.07C Alloy | 44 |
| 15. Tensile Properties of Ta-5W-2.5Mo-0.5Zr-0.07C at 1480 C (2700 F) Showing the Effects of Prior Fabrication History | 48 |
| 16. Recrystallization Temperatures of Ta-5W-2.5Mo-0.5Zr-0.07C for Various Fabrication Conditions | 49 |
| 17. Microstructures and Hardnesses of Ta-5W-2.5Mo-0.5Zr-0.07C for Various Fabrication Conditions | 50 |
| 18. Grain Sizes of Ta-5W-2.5Mo-0.5Zr-0.07C After High-Temperature Annealing | 53 |
| 19. Fabrication Data for Solid Solution Strengthened Tantalum-Base Alloys | 55 |

LIST OF TABLES
(Continued)

| <u>TABLE</u> | <u>Page</u> |
|---|-------------|
| 20. Effect of Temperature on the Bend Ductility of Ta-Mo, Ta-W, and Ta-W-Mo Alloys | 61 |
| 21. Stress-Rupture Properties of Tantalum and Tantalum-Base Alloys at 1480 C (2700 F) | 62 |
| 22. Rupture Strengths of Tantalum and Tantalum-Base Alloys at 1480 C (2700 F) | 66 |
| 23. Stress-Rupture Properties of Tantalum and Tantalum-Base Alloys at 1925 C (3500 F) | 67 |
| 24. Rupture Strengths of Tantalum and Tantalum-Base Alloys at 1925 C (3500 F) | 72 |
| 25. Recrystallization Temperatures for Solid-Solution Tantalum-Base Alloys | 75 |
| 26. Microstructures and Hardnesses of Solid-Solution Tantalum-Base Alloys | 76 |
| 27. Results of Examination of Automatic EB- and TIG-Produced Welds in Tantalum-Base Alloys Selected for Detailed Evaluation . . . | 78 |
| 28. Weld-Bend Ductilities of Tantalum-Base Alloys | 88 |
| 29. Results of Examination of Automatic TIG-Produced Welds in Tantalum-Base Alloys Selected for Detailed Evaluation | 90 |
| 30. Automatic TIG Weld-Bend Ductilities of Ta-5Mo, Ta-12.5W, Ta-5W-2.5Mo, and Ta-10W-2.5Mo | 92 |
| 31. Room-Temperature Tensile Properties of Automatic TIG Weld Properties in Ta-5Mo, Ta-12.5W, Ta-5W-2.5Mo, and Ta-10W-2.5Mo | 94 |
| 32. Fabrication Data for Rhenium- and Ruthenium-Containing Tantalum-Base Alloys | 96 |
| 33. Bend Ductilities of Rhenium- and Ruthenium-Containing Tantalum-Base Alloys at 25 and -195 C (75 and -320 F) | 96 |
| 34. Tensile Properties of Solid-Solution-Strengthened Tantalum-Base Alloys at 1925 C (3500 F). | 97 |
| 35. Recrystallization Temperature for Rhenium- and Ruthenium-Containing Tantalum-Base Alloys | 98 |

LIST OF TABLES
(Continued)

| <u>TABLE</u> | <u>Page</u> |
|---|-------------|
| 36. Microstructures and Hardnesses of Rhenium- and Ruthenium-Containing Tantalum-Base Alloys | 99 |
| 37. Effects of Additions to Tantalum Upon Recrystallization Properties . . | 100 |
| 38. Results and Examination of Manual TIG-Produced Welds in Tantalum-Base Alloys Containing Rhenium and Ruthenium | 100 |
| 39. Manual TIG Weld-Bend Ductilities of Tantalum-Base Alloys Containing Rhenium and Ruthenium | 102 |
| 40. Melting Data for Alloy Compositions Selected for Evaluation | 111 |
| 41. Chemical Analyses of Tantalum-Base Alloys | 114 |
| 42. Automatic TIG Welding Conditions for Dispersion Effectiveness Alloys . | 115 |
| 43. Automatic TIG Welding Conditions for Tantalum-Base Alloys Selected for Detailed Evaluation | 116 |

LIST OF FIGURES

| <u>FIGURE</u> | <u>Page</u> |
|---|-------------|
| 1. Schematic Representation of Stainless Steel and Molybdenum Pack Construction | 9 |
| 2. Specifications for Tensile and Stress-Rupture Specimens | 11 |
| 3. Specifications for Weld-Tensile Specimens | 11 |
| 4. Effect of Rolling and Annealing Temperature on the Microstructure of the Ta-5W-2.5Mo and Ta-5W-2.5Mo-0.5Zr Alloys | 21 |
| 5. Effect of Rolling and Annealing Temperature on the Microstructure of the Ta-5W-2.5Mo-0.07C and Ta-5W-2.5Mo-1Hf-0.07C Alloys | 22 |
| 6. Stress-Rupture Curves for Recrystallized Tantalum, Ta-5W-2.5Mo, and Ta-5W-2.5Mo-0.5Zr-0.07C at 1480 C (2700 F) | 29 |
| 7. Stress-Rupture Curves for Recrystallized Tantalum, Ta-5W-2.5Mo, and Ta-5W-2.5Mo-0.5Zr-0.07C at 1925 C (3500 F) | 30 |
| 8. Fabrication Schedule for the Ta-5W-2.5Mo-0.5Zr-0.07C Alloy | 37 |
| 9. Fabrication Flow Chart for the Ta-5W-2.5Mo-0.5Zr-0.07C Alloy | 38 |
| 10. Effect of Annealing Temperature, Reduction, and Rolling Temperature on Final Sheet Quality of the Ta-5W-2.5Mo-0.5Zr-0.07C Alloy | 39 |
| 11. Close-up of As-Rolled Surface of the Ta-5W-2.5Mo-0.5Zr-0.07C Alloy Showing Mode of Cracking | 42 |
| 12. Typical Microstructure of Cast Ta-5W-2.5Mo-0.5Zr-0.07C | 43 |
| 13. Effect of Rolling and Annealing Temperature on the Microstructure of the Ta-5W-2.5Mo-0.5Zr-0.07C Alloy | 45 |
| 14. Recrystallization Behavior of Ta-5W-2.5Mo-0.5Zr-0.07C Showing the Effect of Process-Annealing Temperature | 51 |
| 15. Effect of Temperature on the Bend Ductility of Recrystallized Ta-Mo, Ta-W, and Ta-W-Mo Alloys | 57 |
| 16. 4T Transition Temperature Versus Alloy Content for Recrystallized Tantalum-Base Alloys Containing Molybdenum and Tungsten | 60 |
| 17. Stress-Rupture Curves for Recrystallized Tantalum and Ta-(2.5,5,10)Mo at 1480 C (2700 F) | 63 |

LIST OF FIGURES
(Continued)

| <u>FIGURE</u> | <u>Page</u> |
|---|-------------|
| 18. Stress-Rupture Curves for Recrystallized Tantalum and Ta-(5,10,20)W at 1480 C (2700 F) | 64 |
| 19. Stress-Rupture Curves for Recrystallized Tantalum, Ta-5W-(2.5,5)Mo, and Ta-(7.5,10)W-2.5Mo at 1480 C (2700 F). | 65 |
| 20. Stress-Rupture Curves for Recrystallized Tantalum and Ta-(2.5,5,7.5)Mo at 1925 C (3500 F) | 68 |
| 21. Stress-Rupture Curves for Recrystallized Tantalum and Ta-(5,10,20)W at 1925 C (3500 F) | 69 |
| 22. Stress-Rupture Curves for Recrystallized Tantalum, Ta-5W-(2.5,5)Mo, and Ta-10W-2.5Mo at 1925 C (3500 F) | 70 |
| 23. Effect of Molybdenum and Tungsten Additions on the Recrystallized 10-Hour Rupture Stress of Tantalum at 1480 C (2700 F) | 73 |
| 24. Effect of Molybdenum and Tungsten Additions on the Recrystallized 10-Hour Rupture Stress of Tantalum at 1925 C (3500 F) | 74 |
| 25. Photomicrographs of EB- and TIG-Welded Solid-Solution Strengthened Tantalum-Base Alloys | 79 |
| 26. Effect of EB and TIG Welding on the Microstructure of Solid-Solution Strengthened Tantalum-Base Alloys | 80 |
| 27. Photomicrograph Showing TIG-Welded Tantalum-Base Alloy Strips Prior to Surface Grinding and Sample Preparation | 91 |
| 28. Effect of Temperature on the Recrystallized Tensile Strength of Tantalum-Base Alloys | 105 |
| 29. Attainable Strength at 1925 C (3500 F) as a Function of Transition Temperature | 106 |
| 30. Effect of Annealing Temperature on the Recrystallization Behavior of Ta-5W-2.5Mo-0.5Zr-0.07C | 117 |

INTRODUCTION

Unalloyed tantalum has a high melting point [2996 C (5425 F)], excellent room-temperature fabricability (i. e., can be rolled to >95 per cent reduction without intermediate annealing), no ductile-to-brittle transition temperature (ductile behavior reported down to 4 K), high density [16.6 g/cm³ (0.600 lb/in.³)], high solubility for both interstitial and substitutional solutes, poor oxidation resistance at temperatures above about 500 C (930 F), and low room-temperature strength (30,000 psi tensile strength, as annealed). Although strength is retained reasonably well at elevated temperature [tensile strength, as annealed, 10,000, 5,000, and 3,000 psi at 1200, 1480, and 1650 C (2190, 2700, and 3000 F), respectively], alloying is needed to provide a useful and competitive structural material for use at high temperatures.

Previous reports^{(1-3)*} have adequately reviewed the state of the art for tantalum and tantalum-base alloys. Recent reports by DMIC⁽⁴⁻¹¹⁾ have reviewed the more significant technological advances of tantalum and serve as a continuing source of up-to-date advances and developments.

The objective of this program was the development of a tantalum alloy or alloys that can be used in structural applications at temperatures in excess of 1370 C (2500 F). The requirements for such an alloy or alloys include good formability (low-temperature ductility) and weldability, and useful strength in the contemplated service-temperature range.

Previous studies, under Contract Nos. AF 33(616)-5668 and AF 33(616)-7688, established binary, ternary, and more complex solid solution alloying effects on selected properties. From these data the most promising major solid-solution additions to tantalum for best retention of strength above 1370 C (2500 F) are tungsten and/or molybdenum. However, further efforts were required to fully establish the high- and low-temperature performance of tantalum alloys containing these additions. To further enhance properties of promising alloys, an investigation of auxiliary dispersion strengthening additions was warranted. The current experimental program included six areas of interrelated and concurrent research and development selected to develop a better understanding of these promising solid solution and solid solution plus dispersion strengthened systems. The areas of research included

*References are included at the end of the report.

Manuscript released by the authors March 1963 for publication as an ASD Technical Documentary Report.

- (1) Dispersion effectiveness
- (2) Fabrication variables
- (3) Transition behavior
- (4) Stress-rupture evaluation
- (5) Welding variables
- (6) Screening alloys.

SUMMARY

The research described in this report was programed to provide the following information:

- (1) The effects of varying additives designed to promote dispersed-phase strengthening in solid solution plus dispersion alloys
- (2) The effects of fabrication variables on the behavior of solid solution plus dispersion strengthened alloys
- (3) In promising solid solution strengthened alloys containing tungsten and/or molybdenum additions
 - (a) The effects of alloying on the ductile-to-brittle transition temperature
 - (b) The effects of alloying on stress-rupture behavior
 - (c) The effects of alloying on weldability.
- (4) The effects of minor auxiliary additions of high-valence elements (rhenium, ruthenium) on properties.

Dispersion Effectiveness

Additions of carbon, zirconium, hafnium, and various combinations of carbon with zirconium or hafnium to a Ta-5W-2.5Mo alloy base were studied. A high-temperature in-process anneal at 2205 C (4000 F) was used for this series of alloys. This seriously impaired the fabricability of the carbon-containing alloys. Carbon-free varieties fabricated well.

All alloys were ductile at room temperature, but all additions effected slight (not serious) loss of ductility at -195 C (-320 F). The liquid-nitrogen ductility of carbon-free alloys appeared to be more uniform and predictable than the carbon-containing varieties.

At 1480 C (2700 F) the dispersed reactive-metal-carbide phase (only the Ta-5W-2.5Mo-0.5Zr-0.07C alloy was investigated at this temperature) was a very effective strengthener in both short-time (tensile) and long-time (stress-rupture) tests. Tensile strength of 38,300 psi and 10-hour rupture strength of 19,200 psi compared very favorably with the respective 23,300-psi and 12,100-psi values for the Ta-5W-2.5Mo alloy base. Whereas recrystallization of the solid solution base decreased its tensile strength from 34,500 psi to 23,300 psi, recrystallization of the Ta-5W-2.5Mo-0.5Zr-0.07C alloy increased its tensile strength from 35,200 to 38,300 psi.

At 1650 C (3000 F), the Ta-5W-2.5Mo-0.5Zr-0.07C alloy was also markedly superior to the base in tensile strength (stress-rupture strength was not evaluated).

At 1925 C (3500 F), all alloys were tensile tested. Carbon additions in the absence of reactive metal additions, were not significantly strengthening. On the other hand, reactive metal additions exhibited pronounced strengthening effects. The Ta-5W-2.5Mo-1Zr alloy exhibited a tensile strength of 15,100 psi, compared to 9,000 psi for the Ta-5W-2.5Mo alloy base. Carbon additions to alloys containing reactive metals lowered their strength significantly; the Ta-5W-2.5Mo-1Zr-0.13C alloy possessed a tensile strength of only 6,500 psi. Although the Ta-5W-2.5Mo-0.5Zr-0.07C alloy was modestly superior to the Ta-5W-2.5Mo base in the short-time tensile test (11,500 versus 9,000 psi), superiority was not maintained much beyond 10 hours in stress-rupture evaluation. Other alloys were not stress-rupture tested.

The various dispersed phases exhibited little effect on recrystallization behavior of the Ta-5W-2.5Mo base.

Welding tests of several reactive-metal-carbide-containing alloys showed these alloys to be completely brittle at room temperature in the as-welded condition. The Ta-5W-2.5Mo alloy was ductile. Carbon-free, reactive-metal-containing alloys were not evaluated.

Effects of Fabrication on a Dispersion-Containing Alloy

The Ta-5W-2.5Mo-0.5Zr-0.07C alloy formed the basis for this investigation. The severe effects of high-temperature [1925 or 2205 C (3500 or 4000 F)] process annealing upon subsequent fabricability were attributed to coarse-grained structures and massive carbide formation formed at, or on cooling from, these high temperatures. A small quantity of material incorporating such solution anneals was obtained for evaluation. Fabricability following a milder, 1650 C (3000 F) process anneal was excellent.

Tensile tests at 1480 C (2700 F) showed

- (1) Final cold-rolling temperature [425 or 980 C (800 or 1800 F)] , amount of final reduction (50 or 75 per cent), or lower process-annealing temperatures [1650 or 1925 C (3000 or 3500 F)] did not significantly affect tensile properties.

- (2) High-temperature [2205 C (4000 F)] process annealing and perhaps subsequent hot rolling [1650 C (3000 F)] significantly increased tensile strength from an average of 35,200 to 41,200 psi for fibered material, or from 38,300 to 51,200 psi for the material in the recrystallized condition.

Unfortunately, the advantages of severe thermal treatment during processing appear untenable because of inherent fabrication difficulties (for sheet production, at least).

Recrystallization parameters were increased by as much as 250 C (450 F) by utilization of the 1925 or 2205 C (3500 or 4000 F) process anneals. Other variables showed no consistent effects upon recrystallization behavior of the Ta-5W-2.5Mo-0.5Zr-0.07C alloy.

Both high- and low-temperature properties of this alloy were seriously degraded by a simulated service exposure of 10 hours at 1925 C (3500 F).

Detailed Behavior of Solid-Solution Alloys

Binary Ta-W, Ta-Mo, and ternary Ta-W-Mo alloys containing up to 20 atomic per cent of the addition elements were fabricated from ingot at temperatures from 25 to 1650 C (75 to 3000 F), as required by alloy content and cast hardness. Fabrication was routine and the resultant alloy strips for evaluation were of excellent quality.

Bend tests to determine transition temperature as a function of alloy content indicated that tantalum will tolerate about 6 weight per cent (10.5 atomic per cent) molybdenum or 12 weight per cent (12 atomic per cent) tungsten without serious impairment of ductility at temperatures approaching absolute zero. At room temperature, tolerance for molybdenum is about 9 weight per cent (15.7 atomic per cent), and for tungsten, about 19 weight per cent (19 atomic per cent). Tolerance for combined tungsten and molybdenum additions was about as would be expected by combining the effects of the individual additions. Thus, tungsten additions are less degrading to low-temperature behavior than are molybdenum additions.

Stress-rupture tests showed pronounced superiority for tungsten additions over molybdenum additions to tantalum at 1480 and 1925 C (2700 and 3500 F). Ten-hour rupture strengths of about 19,000 and 6,500 psi at 1480 and 1925 C (2700 and 3500 F), respectively, should be attainable in binary Ta-W alloys that exhibit good ductility at room temperature. For binary, room-temperature-ductile Ta-Mo alloys, the corresponding maximum strength values would be 14,000 and 4,000 psi. (Tantalum's 10-hour strengths are 4,000 and 750 psi, at these temperatures.)

Welding of these solid solution strengthened alloys raised ductile-to-brittle transition temperatures by 300 to 500 C (540 to 900 F). Electron-beam welding was only slightly superior to TIG welding in improving low-temperature ductility. Weld ductility was markedly improved by homogenization during postweld thermal exposures at 1480 and 1925 C (2700 and 3500 F).

Effects of Rhenium and Ruthenium Additions

The following alloys were studied:

Ta-7.5W-2.5Re

Ta-5W-5Re

Ta-5W-2.5Mo-2.5Re

Ta-5W-2.5Ru.

All alloys were fabricated from cast buttons at 1650 C (3000 F) without difficulty. Atom-for-atom, rhenium and ruthenium additions were far more strengthening in tensile tests at 1925 C (3500 F) than tungsten; however, they were also far more degrading to low-temperature ductility. The beneficial and detrimental effects nullified each other, and considering both high- and low-temperature behavior, the effects of rhenium additions can probably be achieved equally well with more substantial additions of tungsten. The minor ruthenium addition was less attractive.

In brief studies, neither of these additions appeared to reduce the detrimental effects of welding; all alloys were brittle at room temperature in the as-welded condition.

Recrystallization temperatures were increased radically by the minor rhenium and ruthenium additions. The amount of increase agreed well with the expected electronic contribution of these additions related to the effects of tungsten, for example.

It is recommended that:

(1) Studies should be continued on the following alloys:

(a) Ta-(15-20)W

(b) Ta-(10-15W)-0.5Zr-0.07C

(c) Ta-(10-15W)-1Zr.

(2) Studies should be initiated to define and improve the maximum temperature capabilities of alloys containing dispersed phases other than carbides.

(3) The Ta-7.5W-2.5Re alloy showed mild superiority to Ta-W alloys. High-tungsten low-rhenium-containing alloys should be investigated more extensively.

(4) Comprehensive welding-process development studies should be undertaken for high-strength tantalum-base alloys.

GENERAL PROCEDURES

Materials

High-purity tantalum prepared by the electron-beam-melting process was used as base material for these studies. Chemical analyses furnished by the supplier are given in Table 1. As-received cast hardness ranged from 65 to 75 BHN.

Substitutional alloying additions to tantalum were made using highest available-purity alloying elements. Additions were made in the form indicated below:

| <u>Addition</u> | <u>Form</u> |
|-----------------|-----------------|
| Zirconium | Sheet |
| Hafnium | Sheet |
| Molybdenum | Sheet |
| Tungsten | Sheet, rod |
| Rhenium | Powder compacts |
| Ruthenium | Sintered bar |

Interstitial carbon additions were added to the melt as high-purity graphite rod.

Alloy Preparation

Melting

All alloys were prepared as 150-gram ingots by tungsten-electrode-arc melting techniques using a partial atmosphere of helium. Each alloy button was melted at least seven times using 400 to 800 amperes at 30 to 32 volts direct current. Each button was checked for homogeneity by radiographic examination.

Alloy buttons measured about 1-1/2 inches in diameter by about 0.35 inch thick.

Nominal alloy compositions were checked on each alloy button by weighing after arc melting.

Rolling

After the cast hardness was measured, alloy ingots were prepared for fabrication at temperatures from 25 to 1650 C (75 to 3000 F).

Ingots fabricated at room temperature were rolled, using kerosene as a lubricant, to 0.035 to 0.040-inch-thick strip. The strips were annealed in vacuum for 1 hour at 1300 to 1400 C (2370 to 2550 F) at 0.100 inch, thus providing controlled final cold reductions of 60 to 65 per cent.

TABLE 1. ANALYSES OF ELECTRON-BEAM-MELTED TANTALUM

| Element | Impurity Content, ppm | |
|---------|--------------------------|--------|
| | Top | Bottom |
| C | <30 | <30 |
| H | -- | -- |
| N | 40 | 31 |
| O | -- | -- |
| Al | <25 | <25 |
| B | <1 | <1 |
| Cb | 330 | 110 |
| Cd | <5 | <5 |
| Cr | <20 | <20 |
| Cu | <40 | <40 |
| Fe | <40 | <40 |
| Mg | <20 | <20 |
| Mn | <20 | <20 |
| Mo | <20 | <20 |
| Ni | <20 | <20 |
| Pb | <20 | <20 |
| Si | <100 | <100 |
| Sn | <20 | <20 |
| Ti | <20 | <20 |
| V | <20 | <20 |
| W | 220 | 260 |
| Zn | <20 | <20 |

Ingots fabricated at 370 to 425 C (700 to 800 F) were rolled in air to 0.100-inch-thick strip. After the thin surface oxide was removed by pickling (5:2:2 mixture of H_2SO_4 - HNO_3 -HF acids) and any surface defects removed by grinding, the strips were annealed for 1 hour in vacuum at 1400 C (2550 F), and final rolled to 0.040 to 0.045-inch-thick sheet at room temperature. Final reductions were 55 to 65 per cent.

The following procedures were used to prepare alloys for fabrication at 1095 C (2000 F):

- (1) Machine ingots to 1.125 by 1.125 by 0.300-inch rectangular slabs
- (2) Press fit into stainless steel frame (see Figure 1)
- (3) Assemble pack using a small amount of parting compound (Cr_2O_3 mixed with water to brushing consistency) between molybdenum shims and stainless steel cover plates
- (4) Weld seven edges under helium; weld final edge by electron-beam welding, thus insuring a vacuum of less than 5×10^{-4} mm Hg within the stainless steel pack.

Alloys were then rolled in the stainless steel packs at 1095 C (2000 F), using an argon preheating atmosphere to minimize the severity of contamination should pack failure occur, to 0.095 to 0.100-inch-thick strip. The alloys were stripped from the packs, conditioned by pickling (5:2:2 mixture of H_2SO_4 - HNO_3 -HF acids) and grinding, vacuum annealed for 1 hour at 1500 C (2730 F), and final rolled 50 to 55 per cent at 260 to 425 C (500 to 800 F), unprotected in air.

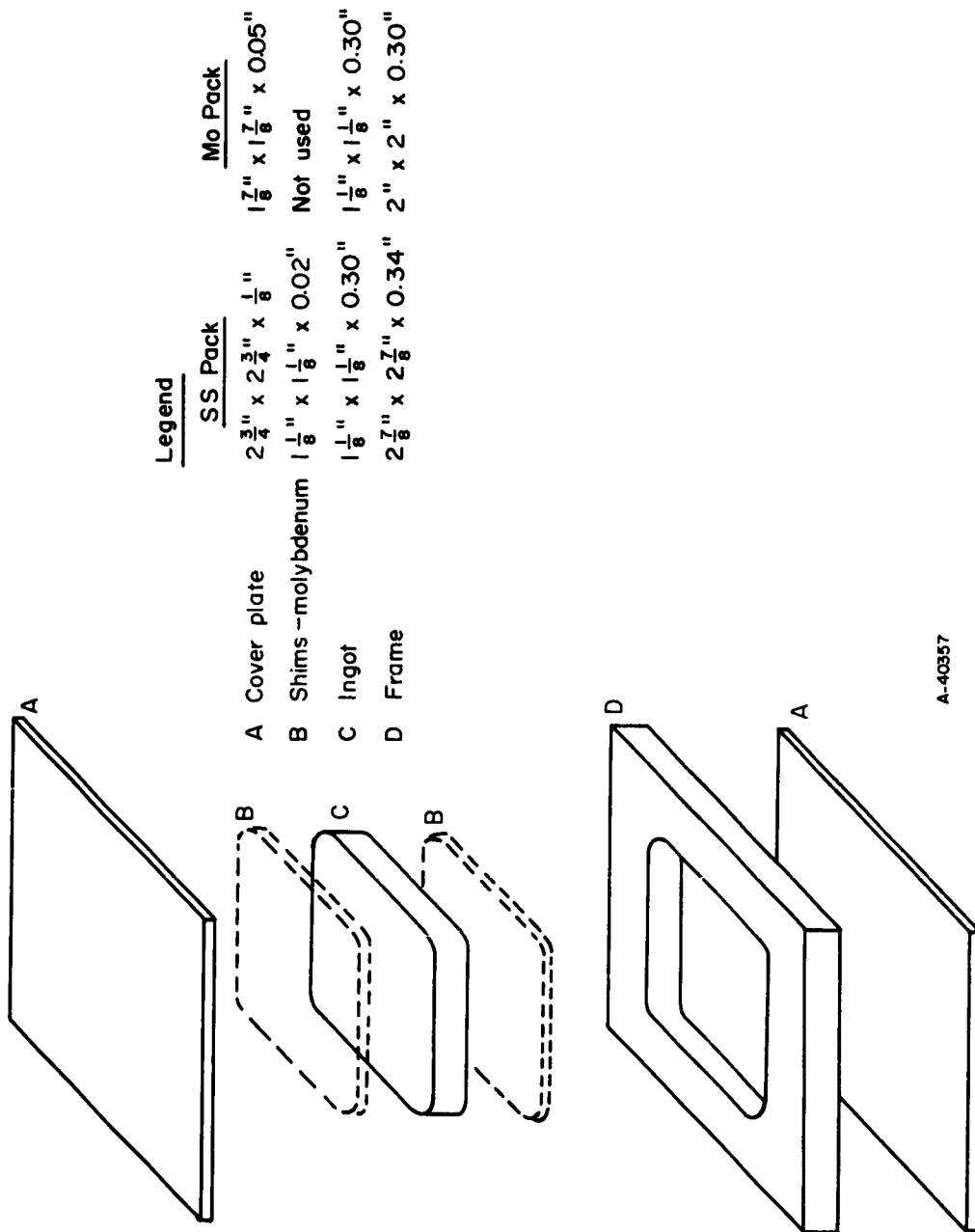
The following procedures were used to prepare alloys for fabrication at 1650 C (3000 F):

- (1) Machine ingots to 1.125 by 1.125 by 0.300-inch rectangular slabs
- (2) Press fit ingots into molybdenum frame (see Figure 1)
- (3) Weld seven edges under helium; weld final edge by electron-beam welding, thus insuring a vacuum of less than 5×10^{-4} mm Hg within the molybdenum pack.

These packs were rolled at 1650 C (3000 F), using a hydrogen preheating atmosphere, to 0.075 to 0.180-inch-thick strip. Alloy strips were leached from the molybdenum packs using warm [about 95 C (200 F)] HNO_3 acid, surface conditioned by grinding (to final sheet thickness of about 0.040-inch strip for a number of alloys rolled 75 per cent from the button ingot - other alloys were reduced further at lower temperatures), annealed in vacuum for 1 hour at 1650 to 2205 C (3000 to 4000 F), and where appropriate, final rolled 50 to 65 per cent to 0.040-inch-thick strip at 425 to 980 C (800 to 1800 F).

Heat Treatment

Test specimens were annealed in vacuum at 1100 to 2205 C (2019 to 4000 F) for 1 to 10 hours. Pressures were always below $1 \text{ by } 10^{-4}$ mm Hg. Specimens were



A-40357

FIGURE 1. SCHEMATIC REPRESENTATION OF STAINLESS STEEL AND MOLYBDENUM PACK CONSTRUCTION

heated by a resistance tantalum heater element with manual temperature control. An optical pyrometer was used (employing the proper corrections) to measure the specimen temperature to an accuracy of about ± 5 C (± 10 F).

Specimen Preparation

Specimens to study recrystallization behavior were cut to approximately 3/8 by 1/8-inch size from 0.035 to 0.045-inch-thick strip with the long edge (longitudinal) parallel to the rolling direction. Metallographic observations and hardness measurements were made on the longitudinal sections.

Bend-test specimens were cut to a 1 by 1/4-inch size from 0.035 to 0.045-inch-thick strip with the length dimension in the rolling direction. All surfaces were ground through 240X paper; the tension side was ground through 600X paper. Specimens were then recrystallized prior to testing.

Weld-bend specimens for screening evaluation were prepared by butting shoulders of tested tensile specimens together and laying a weld bead parallel to the prior rolling direction, using manual, inert-gas, tungsten-arc (TIG) welding techniques. After visual and radiographic examination the top and bottom sheet surfaces were ground using a 60X wheel, then all surfaces were ground through 240X paper; the tension side was ground through 600X paper. Specimens measured about 3/4 by 1/4 by 0.025 to 0.035 inch.

Specimens for more detailed weld-bend-ductility evaluation were prepared by cutting 1/2-inch wide strips (3 to 5 inches long) from 0.045 to 0.050-inch-thick recrystallized sheet. The long dimension of each strip was parallel to the rolling direction. Prior to welding, the strips were cleaned with 240X paper and degreased in acetone. Two of the strips were butted together side-to-side and clamped in a jig for each weld. These simple butt joints were welded by the inert-gas, tungsten-arc (TIG) and the electron-beam (EB) processes. After visual and radiographic examination the top and bottom sheet surfaces were wheel ground using a 60X wheel, then all surfaces were ground through 240X paper; the tension side was ground through 600X paper. Specimens were cut to about 1 by 3/4 inch from the 0.030 to 0.040-inch-thick sheet to provide a test sample. The bend axis traversed the weld metal, heat-affected zone, and base metal.

Tensile and stress-rupture specimens were cut from 0.035 to 0.045-inch-thick strip with the tension axis parallel to the rolling direction. Specifications are shown in Figure 2. The smaller specimen design was used when a shortage of fabricated sheet existed.

Weld tensile specimens were cut from 60X wheel-ground 0.030 to 0.040-inch-thick strip with tension axes parallel to the rolling direction and weld direction. Specifications are shown in Figure 3. The reduced section encompassed weld metal, heat-affected zone, and base metal.



FIGURE 3. SPECIFICATIONS FOR WELD-TENSILE SPECIMENS

Metallographic Techniques

Specimens for metallographic study were mounted in Bakelite or Epon, ground through 600X paper, and mechanically polished, finishing on a high-speed wheel with a mixture of alumina and chromic acid. The specimens were chemically etched by dissolving 30 to 40 grams of $\text{NH}_4\text{F} \cdot \text{HF}$ in 50 to 70 milliliters HNO_3 and 100 milliliters H_2O .

Mechanical Testing

Hardness values were measured using the Vickers hardness test and a 10-kilogram load. The values reported are the average of five impressions on each specimen.

Specimens were tested for bend ductility over the temperature range -195 to 305 C (-320 to 600 F) in the recrystallized condition. Each specimen was bent by hand using an arbor press through a progressively sharper die until evidence of cracking appeared. Die radii used are listed below:

| Die Radius, in. | Decimal Equivalent, in. | Die Radius, in. | Decimal Equivalent, in. |
|-----------------------|-------------------------------|-----------------------|-------------------------------|
| 1-1/2 | 1.5 | 3/32 | 0.0937 |
| 3/4 | 0.75 | 1/16 | 0.0625 |
| 3/8 | 0.375 | 3/64 | 0.0468 |
| 1/4 | 0.25 | 1/32 | 0.0312 |
| 3/16 | 0.1875 | 1/64 | 0.0156 |
| 1/8 | 0.125 | Sharp | -- |

The bend ductility value, T, was calculated from

$$T = \frac{r}{t},$$

where

T = bend ductility

r = radius of last good die before evidence of cracking appears, in.

t = specimen thickness, in.

Weld-bend specimens were tested for bend ductility at 25 to 315 C (75 to 600 F), using the same procedure outlined above, by bending on an axis perpendicular to the weld until evidence of cracking appeared.

Room-temperature tensile tests were conducted using conventional hydraulic testing machines. Load-strain curves were recorded autographically from strain gages attached to the specimen. A crosshead speed of 0.025 inch per minute was used for the entire test to fracture (corresponding to an approximate strain rate of 0.05 inch per inch per minute for the 1/2-inch reduced section design which was used for all room-temperature tensile tests).

For room-temperature weld tensile specimens, a crosshead speed was 0.01 inch per minute through 0.6 per cent yield strength and 0.025 inch per minute for the remainder of the test to fracture (corresponding to approximate strain rates of 0.02 and 0.05 inch per inch per minute, respectively, for a 1/2-inch reduced section).

A detailed description of the test units used for high-temperature [1480 to 1925 C (2700 to 3500 F)] tensile and stress-rupture tests has previously been reported^(3,13). Tensile loads were applied to the specimen by a variable-speed screw-driven crosshead.

Tests for prior tantalum alloy development programs at Battelle have utilized crosshead movements of approximately 0.01 inch per minute through the 0.2 per cent offset yield strength, and 0.05 inch per minute to fracture, for a 1-1/4-inch reduced gage section. However, the MAB⁽¹²⁾ recommends that a strain rate of 0.05 ± 0.02 inch per inch per minute be maintained from zero strain to fracture. To standardize test procedure in accord with MAB recommendations, all high-temperature tensile tests in the current program were conducted at a constant crosshead movement for the entire test. For the 1-1/4-inch reduced gage section a constant crosshead movement of 0.05 inch per minute was used - this corresponds to an initial nominal strain rate of 0.04 inch per inch per minute. For the smaller test samples, 1/2-inch reduced gage section, a constant crosshead movement of 0.02 inch per minute was used - this also corresponds to an initial nominal strain rate of 0.04 inch per inch per minute. It should be noted that the strain rate was nominal and changed as the specimen elongated; however, the nominal strain rate was well within the 0.05 ± 0.02 inch per inch per minute tolerance.

It was also recognized that conducting high-temperature tensile tests in this manner may have resulted in somewhat higher yield strength relative to past studies since a higher strain rate resulted from the increased crosshead movement (0.01 versus 0.05 inch per minute for the 1-1/4-inch reduced gage section). Tensile strengths should be comparable, however. [Proof tests on recrystallized Ta-10W sheet specimens showed the tensile strength at 1925 C (3500 F; 10,000 psi) to be independent of the above testing technique variation. Yield strength was affected slightly, as expected.]

Loads were measured by a stainless steel-ring load cell, mounted inside the vacuum chamber and shielded from the heated zone. Strain-gage signals fed into a Sanborn 150 recorder provided a load-time record for each test.

Specimens were heated by a tantalum resistance heater element with manual temperature control. An optical pyrometer was used (employing the proper corrections) to measure the specimen temperature to an accuracy of about ± 5 C (± 10 F).

During heating and testing at temperatures through 1925 C (3500 F) pressures less than 1×10^{-4} mm Hg were maintained. All test specimens were heated to test temperature in approximately 45 minutes [for 1925 C (3500 F) tests] or less.

Rupture-test equipment and procedures were similar to those described above for tensile testing; however, loads are applied through a lever-magnified dead-weight loading system rather than the screw-driven crosshead. In all cases the tensile strength at 1480 and 1925 C (2700 and 3500 F) was used as a guide to estimate the stress levels necessary to cause rupture in 1 to 10 hours.

PROGRAM DESIGN AND ALLOY SELECTION

Past work^(2,3) has shown that molybdenum and tungsten are the most promising major solid solution additions to tantalum for best retention of strength above 1370 C (2500 F). Minor additions of rhenium and ruthenium were also quite attractive for significant improvements in high-temperature properties. Auxiliary dispersion strengthening additions, such as reactive metals (Zr, Hf) and carbon, appeared to be an area where more detailed investigations were warranted. Based on the promising results of past studies, the current program was designed to better establish the high- and low-temperature behavior of the more promising solid solution additions (W, Mo, Re, Ru) as well as to determine the effectiveness of auxiliary dispersion strengthening additions (Zr, Hf, C). The current program included six areas of interrelated research and development, selected to better establish solid solution and solid solution plus dispersion strengthening behavior in tantalum-base alloys, as indicated below:

- (1) Dispersion Effectiveness - evaluate the effectiveness of various dispersions on the strength, ductility, weldability, and recrystallization behavior of a Ta-5W-2.5Mo base.
- (2) Fabrication Variables - investigate the effects of processing variables, including temperature of fabrication, annealing temperature, and amount of reduction, and attempt to define fabrication history for the development of optimum properties for the Ta-5W-2.5Mo-0.5Zr-0.07C alloy.
- (3) Transition Behavior - establish the low-temperature bend ductility transition behavior for the promising alloy additions of tungsten and molybdenum.
- (4) Stress-Rupture Evaluation - determine the high-temperature [1480 and 1925 C (2700 and 3500 F)] stress-rupture behavior of binary and ternary alloys containing tungsten and molybdenum.
- (5) Welding Variables - examine automatic TIG and EB welding techniques to determine the effects of welding process variables on the weld-bend ductility behavior for tantalum alloys containing tungsten and molybdenum. In addition, the effects of high-temperature [1480 and 1925 C (2700 and 3500 F)] postweld thermal exposure were studied.
- (6) Screening Alloys - minor additions of Group VII-A (Re) and Group VIII-A (Ru) metals were evaluated for their effects on 1925 C (3500 F) strength, low-temperature ductility, weldability, and recrystallization behavior.

Table 2 lists the 69 button ingots designed to provide sufficient material for anticipated tests to cover the areas of research listed and described above. Several of the alloys selected to study dispersion effectiveness failed during fabrication, as described in the "Dispersion Effectiveness" section of this report, and several of the more critical compositions were selected for repeat fabrication. These alloys are covered in Table 3.

TABLE 2. ALLOY COMPOSITIONS SELECTED FOR EVALUATION

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Composition (Balance Tantalum), atomic per cent | Number of Ingots ^(a) |
|---|---|------------------------------------|
| <u>Dispersion Effectiveness</u> | | |
| 5W-2.5Mo | 4.8W-4.6Mo | 1 |
| 5W-2.5Mo-0.07C | 4.7W-4.6Mo-1.1C | 1 |
| 5W-2.5Mo-0.13C | 4.7W-4.5Mo-1.9C | 1 |
| 5W-2.5Mo-1Hf | 4.8W-4.6Mo-1.1Hf | 1 |
| 5W-2.5Mo-2Hf | 4.8W-4.6Mo-2.0Hf | 1 |
| 5W-2.5Mo-0.5Zr | 4.8W-4.6Mo-0.9Zr | 1 |
| 5W-2.5Mo-1Zr | 4.7W-4.6Mo-1.9Zr | 1 |
| 5W-2.5Mo-1Hf-0.07C | 4.7W-4.6Mo-1.1Hf-1.1C | 1 |
| 5W-2.5Mo-0.5Zr-0.07C | 4.7W-4.5Mo-0.9Zr-1.0C | 2 |
| 5W-2.5Mo-0.5Zr-0.13C | 4.7W-4.5Mo-0.9Zr-1.9C | 1 |
| 5W-2.5Mo-1Zr-0.07C | 4.7W-4.5Mo-1.9Zr-1.0C | 1 |
| 5W-2.5Mo-1Zr-0.13C | 4.7W-4.5Mo-1.9Zr-1.9C | 2 |
| <u>Fabrication Variables</u> | | |
| 5W-2.5Mo-0.5Zr-0.07C | 4.7W-4.5Mo-0.9Zr-1.0C | 7 |
| <u>Transition Behavior</u> | | |
| 2.5Mo | 4.6Mo | 1 |
| 5Mo | 9.0Mo | 1 |
| 7.5Mo | 13.2Mo | 1 |
| 10Mo | 17.3Mo | 1 |
| 10W | 9.8W | 1 |
| 15W | 14.9W | 1 |
| 20W | 19.8W | 1 |
| 5.2W-2.7Mo | 5.0W-5.0Mo | 1 |
| 7.9W-4.1Mo | 7.5W-7.5Mo | 1 |
| 10.6W-5.6Mo | 10.0W-10.0Mo | 1 |
| <u>Stress-Rupture Evaluation</u> | | |
| 100Ta | 100Ta | 1 |
| 2.5Mo | 4.6Mo | 2 |
| 5Mo | 9.0Mo | 2 |
| 7.5Mo | 13.2Mo | 1 |
| 10Mo | 17.3Mo | 1 |
| 5W | 4.9W | 1 |
| 10W | 9.8W | 1 |
| 20W | 19.8W | 2 |
| 5W-2.5Mo | 4.8W-4.6Mo | 1 |
| 5W-5Mo | 4.6W-9.0Mo | 2 |
| 5W-7.5Mo | 4.6W-13.2Mo | 2 |

TABLE 2. (Continued)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Composition (Balance Tantalum), atomic per cent | Number of Ingots ^(a) |
|---|---|------------------------------------|
| 10W-2.5Mo | 9.6W-4.6Mo | 2 |
| 10W-5Mo | 9.4W-9.0Mo | 2 |
| 15W-2.5Mo | 14.5W-4.6Mo | 2 |
| <u>Welding Variables</u> | | |
| 5Mo | 9.0Mo | 3 |
| 12.5W | 12.3W | 3 |
| 5W-2.5Mo | 4.8W-4.6Mo | 3 |
| 10W-2.5Mo | 9.6W-4.6Mo | 3 |
| <u>Screening Alloys</u> | | |
| 5W-2.5Ru | 4.8W-4.4Ru | 1 |
| 5W-5Re | 4.9W-4.9Re | 1 |
| 5W-2.5Mo-2.5Re | 4.8W-4.6Mo-2.3Re | 1 |
| 7.5W-2.5Re | 7.4W-2.4Re | 1 |

(a) 150-gram ingots.

TABLE 3. REPEAT ALLOY COMPOSITIONS SELECTED FOR
EVALUATION OF DISPERSION EFFECTIVENESS

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Composition (Balance Tantalum), atomic per cent | Number of Ingots ^(a) |
|---|---|------------------------------------|
| <u>Dispersion Effectiveness</u> | | |
| 5W-2.5Mo-1Hf-0.07C | 4.7W-4.6Mo-1.1Hf-1.1C | 2 |
| 5W-2.5Mo-0.5Zr-0.07C | 4.7W-4.5Mo-0.9Zr-1.0C | 6 |
| 5W-2.5Mo-0.5Zr-0.13C | 4.7W-4.5Mo-0.9Zr-1.9C | 1 |
| 5W-2.5Mo-1Zr-0.07C | 4.7W-4.5Mo-1.9Zr-1.0C | 2 |
| 5W-2.5Mo-1Zr-0.13C | 4.7W-4.5Mo-1.9Zr-1.9C | 1 |

(a) 150-gram ingots.

EXPERIMENTAL RESULTS

Based on radiographic examination, weight-change data, and hardness measurements, all 45 compositions outlined in Table 2 were successfully prepared. Melting data for these alloys are given in Appendix I. Hardness and weight-change data are consistent with previous work and indicate good compositional control for all alloys. Seven of the solid solution plus dispersion strengthened alloys picked up slight contamination from the tungsten electrode and were reprepared.

In addition to weight balance as an indication of conformance of actual to nominal composition, chemical analyses were made on fabricated strip of many compositions involving minor reactive metal (0.5 to 2 per cent Zr or Hf) or interstitial (0.7 to 0.13 per cent C) additions. Analytical data are recorded in Appendix I. In all cases, carbon and reactive metal additions were maintained at the desired level. Oxygen contents were low (3 to 28 ppm) suggesting some purification during melting or processing these Ta-W-Mo-base alloys. Nitrogen and hydrogen contents were normal. Tungsten and molybdenum contents were consistent in these alloys.

These analyses showed the excellent control of both interstitial and substitutional elements in the tantalum-base alloys studied and confirmed expectations based on weight-change data resulting from the melting operation (± 0.2 per cent of all major additions).

Because of divergence of objectives and evaluation techniques involved in the various alloys prepared for this study, the research results are presented in this report in accordance with the specific objectives of the following phases of investigation:

- (1) Dispersion effectiveness
- (2) Fabrication effects
- (3) Detailed behavior of solid solution alloys
 - (a) Transition behavior
 - (b) Stress-rupture behavior
 - (c) Weldability
- (4) Effects of rhenium and ruthenium additions.

Dispersion Effectiveness

In this phase of investigation, the Ta-5W-2.5Mo alloy formed the basis for investigation. As shown in Table 2, the effects of individual additions of carbon, zirconium, and hafnium, each at approximate levels of 1 and 2 atomic per cent, were investigated. Combined additions of zirconium and carbon were studied at the 1 and 2 mol (ZrC) per cent level in stoichiometric balance, at the 1 per cent level with excessive carbon, and at the 2 per cent level with a deficiency in carbon. Combined

additions of hafnium and carbon were studied at the 1 mol (HfC) per cent level, in stoichiometric balance only.

Fabrication

Although method of fabrication was not intended to be a variable, inability to satisfactorily fabricate certain of these compositions by the initially selected route required the inclusion of different fabrication techniques in different materials as discussed below.

The initial fabrication procedure for the alloys selected to study dispersion effectiveness in a Ta-5W-2.5Mo alloy base included a high-temperature intermediate solution anneal [2205 C (4000 F)] to take advantage of carbide solution and subsequent age and/or work-hardening characteristics developed during final rolling:

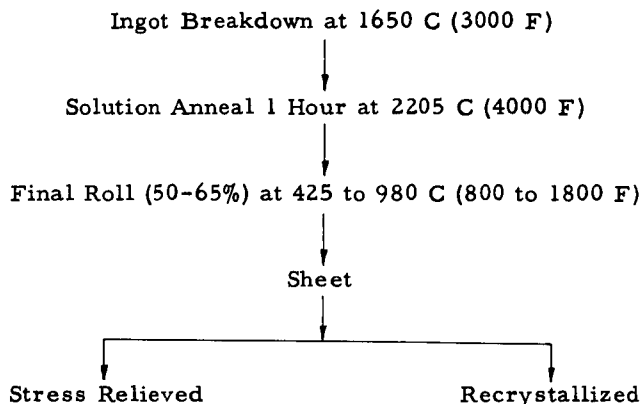


Table 4 presents the fabrication results for all materials prepared for the study of dispersion effectiveness. Primary fabrication of ingots at 1650 C (3000 F) was accomplished without difficulty. Typical microstructures of selected alloys after primary fabrication and the 2205 C (4000 F) process anneal are shown in Figures 4 and 5. As shown, the Ta-5W-2.5Mo base composition is essentially single phase in the absence of a high-temperature anneal, but develops a small amount of inter- and intragranularly dispersed phase upon annealing at (or cooling from) high temperatures. The addition of a reactive metal modestly increases the amount of second phase. Carbon additions, however, either with or without concurrent reactive metal additions, radically increase the amount of second phase, which appears in Figure 5 primarily in a Widmanstätten pattern and in massive form at grain boundaries.

Final rolling at 425 to 980 C (800 to 1800 F) following intermediate annealing at 2205 C (4000 F), indicated superior fabricability for the Ta-5W-2.5Mo alloy compared with carbon-containing alloys; the addition of 0.07 per cent carbon to Ta-5W-2.5Mo caused slight but significant edge cracking during sheet rolling. Increasing the carbon content to 0.13 per cent further degraded fabricability. Small zirconium additions (up to 1 per cent) or hafnium additions (up to 2 per cent) were not particularly degrading to fabricability; however, combined zirconium or hafnium and carbon additions (alloys containing about 0.5 per cent zirconium and 0.07 per cent carbon, etc.) were markedly detrimental, and appeared to have about the same effects on the fabricability of the

TABLE 4. FABRICATION DATA FOR DISPERSION-EFFECTIVENESS ALLOYS

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Cast Hardness (a), VHN | Initial Rolling Tempera- ture(b) | | Quality of Strip (c) | Intermediate Annealing Tempera- ture | | Hardness(a), VHN | | Final Rolling Temperature(b) | | Final Reduction, per cent | Quality of Strip (c) |
|---|-------------------|------------------------------|---|------|-------------------------|---|------|---------------------|----------|---------------------------------|----------|---------------------------------|-------------------------|
| | | | C | F | | C | F | Rolled | Annealed | C | F | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| Initial Studies | | | | | | | | | | | | | |
| 5W -2. 5Mo | 268G | 213 | 1650 | 3000 | Excellent | 2205 | 4000 | 276 | 254 | 425 | 800 | 65 | Excellent |
| 5W -2. 5Mo-0. 07C | 311A | 272 | 1650 | 3000 | Excellent | 2205 | 4000 | 342 | 266 | 480 | 900 | 60 | Good(d) |
| 5W -2. 5Mo-0. 13C | 312A | 309 | 1650 | 3000 | Excellent | 2205 | 4000 | 357 | 272 | 480/980 | 900/1800 | 30/30 | Fair/Fair(e) |
| 5W -2. 5Mo-1Hf | 319 | 228 | 1650 | 3000 | Excellent | 2205 | 4000 | 302 | 276 | 980 | 1800 | 65 | Excellent |
| 5W -2. 5Mo-2Hf | 320 | 242 | 1650 | 3000 | Excellent | 2205 | 4000 | 306 | 289 | 980 | 1800 | 65 | Excellent |
| 5W -2. 5Mo-0. 5Zr | 313 | 222 | 1650 | 3000 | Excellent | 2205 | 4000 | 309 | 272 | 425 | 800 | 65 | Excellent |
| 5W -2. 5Mo-1Zr | 314 | 232 | 1650 | 3000 | Excellent | 2205 | 4000 | 319 | 292 | 480 | 900 | 60 | Good |
| 5W -2. 5Mo-1Hf-0. 07C | 321 | 289 | 1650 | 3000 | Excellent | 2205 | 4000 | 357 | 285 | 980 | 1800 | 50 | Fair |
| 5W -2. 5Mo-0. 5Zr-0. 07C | 315I | 289 | 1650 | 3000 | Excellent | 2205 | 4000 | 376 | 276 | 980 | 1800 | 50 | Poor |
| 5W -2. 5Mo-0. 5Zr-0. 07C | 315J | 283 | 1650 | 3000 | Excellent | 2205 | 4000 | 373 | 285 | 980 | 1800 | 50 | Poor |
| 5W -2. 5Mo-0. 5Zr-0. 13C | 318 | 333 | 1650 | 3000 | Excellent | 2205 | 4000 | 376 | 292 | 980 | 1800 | 50 | Poor |
| 5W -2. 5Mo-1Zr-0. 07C | 317A | 299 | 1650 | 3000 | Excellent | 2205 | 4000 | 387 | 309 | 980 | 1800 | 50 | Poor |
| 5W -2. 5Mo-1Zr-0. 13C | 316 | 348 | 1650 | 3000 | Excellent | 2205 | 4000 | 413 | 312 | 980 | 1800 | 50 | Poor |
| 5W -2. 5Mo-1Zr-0. 13C | 316A | 357 | 1650 | 3000 | Excellent | 2205 | 4000 | 397 | 317 | 980 | 1800 | 50 | Poor |
| Repeat Studies | | | | | | | | | | | | | |
| 5W -2. 5Mo-Hf-0. 07C | 321A | 276 | 1650 | 3000 | Excellent | 1650 | 3000 | -- | -- | 425 | 800 | 50 | Excellent |
| 5W -2. 5Mo-Hf-0. 07C | 321B | 276 | 1650 | 3000 | Excellent | 1650 | 3000 | -- | -- | 425 | 800 | 50 | Excellent |

TABLE 4. (Continued)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Cast Hardness ^(a) , VHN | Initial Rolling Tempera- ture ^(b) | | Quality of Strip ^(c) | Intermediate Annealing Tempera- ture | | Hardness ^(a) , VHN | | Final Rolling Temperature ^(b) | | Final Reduction, per cent | Quality of Strip ^(c) |
|---|-------------------|--|---|------|------------------------------------|---|------|----------------------------------|----------|---|-----|---------------------------------|------------------------------------|
| | | | C | F | | C | F | Rolled | Annealed | C | F | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| Repeat Studies (Continued) | | | | | | | | | | | | | |
| 5W-2, 5Mo-0, 5Zr-0, 0.07C | 315L | 281 | 1650 | 3000 | Excellent | 1650 | 3000 | -- | -- | 425 | 800 | 50 | Excellent |
| 5W-2, 5Mo-0, 5Zr-0, 0.07C | 315M | 276 | 1650 | 3000 | Excellent | 1650 | 3000 | -- | -- | 425 | 800 | 50 | Excellent |
| 5W-2, 5Mo-0, 5Zr-0, 0.07C | 315N | 276 | 1650 | 3000 | Excellent | 1650 | 3000 | -- | -- | 425 | 800 | 50 | Excellent |
| 5W-2, 5Mo-0, 5Zr-0, 0.07C | 315O | 279 | 1650 | 3000 | Excellent | 1650 | 3000 | -- | -- | 425 | 800 | 50 | Excellent |
| 5W-2, 5Mo-0, 5Zr-0, 0.07C | 315P | 283 | 1650 | 3000 | Excellent | 1650 | 3000 | -- | -- | 425 | 800 | 50 | Excellent |
| 5W-2, 5Mo-0, 5Zr-0, 0.07C | 315Q | 274 | 1650 | 3000 | Excellent | 1650 | 3000 | -- | -- | 425 | 800 | 50 | Excellent |
| 5W-2, 5Mo-0, 5Zr-0, 0.13C | 318A | 314 | 1650 | 3000 | -- | -- | -- | -- | -- | -- | -- | 75 | Excellent |
| 5W-2, 5Mo-1Zr-0, 0.07C | 317B | 289 | 1650 | 3000 | -- | -- | -- | -- | -- | -- | -- | 75 | Excellent |
| 5W-2, 5Mo-1Zr-0, 0.07C | 317C | 285 | 1650 | 3000 | -- | -- | -- | -- | -- | -- | -- | 75 | Excellent |
| 5W-2, 5Mo-1Zr-0, 0.13C | 316B | 325 | 1650 | 3000 | -- | -- | -- | -- | -- | -- | -- | 75 | Excellent |

(a) Hardness values are the average of five impressions using a 10-kg load.

(b) Alloys rolled at 425 to 480 C (800 to 900 F) unprotected in air. Alloys rolled at 980C (1800 F) in evacuated stainless steel packs. Alloys rolled at 1650 C (3000 F) in evacuated molybdenum packs.

(c) Excellent - no cracking of edges or surface

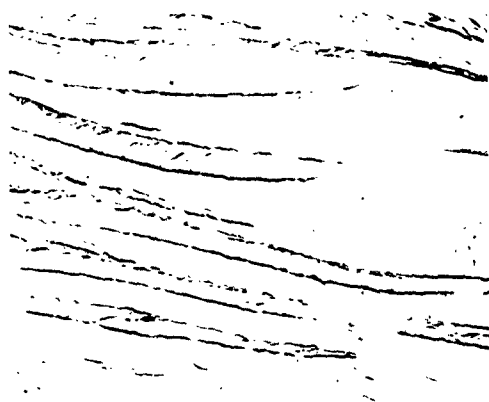
Good - slight cracking of edges and surface

Fair - considerable cracking of edges and surface

Poor - extensive cracking throughout specimen.

(d) Cracking confined to edges only.

(e) Considerable cracking along edges and grain boundaries after 30 per cent reduction at 480 C (900 F). Alloy strip conditioned by machining prior to 30 per cent reduction at 980 C (1800 F).



250X

Rolled 65% at 1650 C
(3000 F); 276 VHN.

N93203



250X

Annealed 1 hour at 2205 C
(4000 F); 254 VHN.

N93204

a. Ta-5W-2.5Mo (Specimen 268G)



250X

Rolled 65% at 1650 C
(3000 F); 309 VHN.

N93209



250X

Annealed 1 hour at 2205 C
(4000 F); 272 VHN.

N93210

b. Ta-5W-2.5Mo-0.5Zr (Specimen 313)

FIGURE 4. EFFECT OF ROLLING AND ANNEALING TEMPERATURE ON THE MICROSTRUCTURE OF THE Ta-5W-2.5Mo AND Ta-5W-2.5Mo-0.5Zr ALLOYS

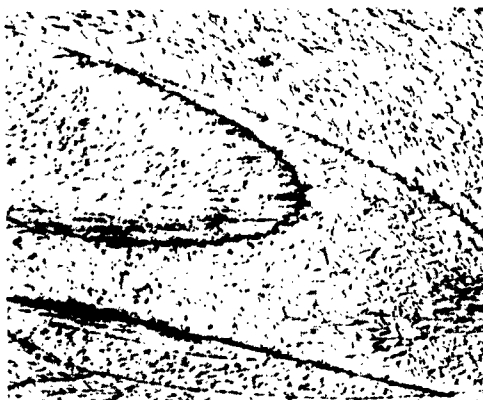


Rolled 65% at 1650 C
(3000 F); 342 VHN.

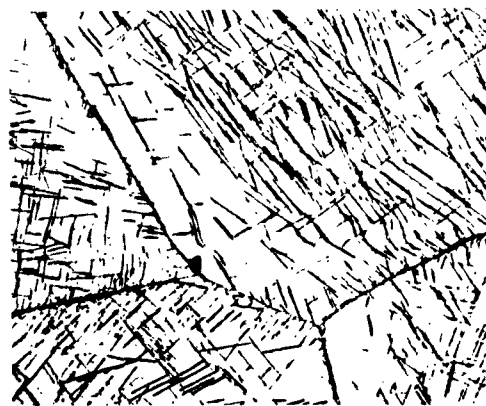


Annealed 1 hour at 2205 C
(4000 F); 266 VHN.

a. Ta-5W-2.5Mo-0.07C (Specimen 311A)



Rolled 65% at 1650 C
(3000 F); 357 VHN.



Annealed 1 hour at 2205 C
(4000 F); 285 VHN.

b. Ta-5W-2.5Mo-1Hf-0.07C (Specimen 321)

FIGURE 5. EFFECT OF ROLLING AND ANNEALING TEMPERATURE ON THE MICROSTRUCTURE OF THE Ta-5W-2.5Mo-0.07C AND Ta-5W-2.5Mo-1Hf-0.07C ALLOYS

Ta-5W-2.5Mo alloy as 0.13 per cent carbon. Because of failure of most of the reactive-metal-plus-carbon-containing alloys to fabricate satisfactorily following the high-temperature anneal, several ingots of these compositions were reprepared and fabricated by a schedule wherein the 2205 C (4000 F) process anneal was omitted or replaced with a 1650 C (3000 F) anneal. In no case did the resultant structures show the coarse grains and profuse, massive Widmanstätten structure illustrated in Figure 5. All repeat compositions fabricated excellently to sheet.

Evaluation

The effectiveness of the various dispersion-forming additions to Ta-5W-2.5Mo was evaluated in various mechanical tests and by recrystallization performance. Principal among the mechanical tests were bend tests (on all but the Ta-5W-2.5Mo-0.5Zr-0.13C) at room and liquid-nitrogen temperatures and tensile tests at 1925 C (3500 F). Tensile tests at room temperature, 1430 C (2700 F), and 1650 C (3000 F), and stress-rupture tests at 1480 and 1925 C (2700 and 3500 F) were also conducted, but were severely restricted by material availability.

Unless otherwise noted, all materials were fully recrystallized prior to mechanical evaluation.

Bend Tests. The bend ductilities for the various dispersion-effectiveness alloys at 25 and -195 C (75 and -320 F) are given in Table 5. None of the additions to the Ta-5W-2.5Mo were detrimental to bend ductilities at room temperature. However, all exhibited some degradation at -195 C (-320 F). The reactive metal additions at the 1 atomic per cent level (0.5Zr, 1Hf) were not significantly detrimental; but at the higher level were definitely degrading, although the effects were not severe. Material containing 1 atomic per cent carbon exhibited erratic behavior at the lower temperature. Stoichiometric molar ZrC or HfC additions at the 1 mol per cent level also showed erratic behavior in one of two cases. The carbon-deficient Ta-5W-2.5Mo-1Zr-0.07C alloy exhibited about the same liquid-nitrogen ductility as the carbon-free Ta-5W-2.5Mo-1Zr alloy. No carbon-excess reactive metal-containing material was available for testing.

These results suggest that minor reactive metal additions may be tolerable and are predictable regarding their effects on low-temperature ductility. Carbon additions, either with or without concurrent reactive metal additions require further study, but an aura of caution is suggested by the data of Table 5.

Tensile Tests. Table 6 gives room-temperature tensile properties of three recrystallized tantalum-base alloys containing dispersed carbide phases. Although the metallurgical condition of the base composition, Ta-5W-2.5Mo, was partially fibered, the addition of 0.5-1 per cent zirconium or 1 per cent hafnium in the presence of 0.07 per cent carbon seems to have little effect on tensile properties. For example, strength levels for these carbon containing alloys were about the same as that of recrystallized solid solution Ta-10W alloy⁽³⁾, which contains about the same level of strengthener as Ta-5W-2.5Mo. Data for Specimens 315P and 315Q show the degree of scatter between two different button ingots of the same nominal composition fabricated in the same way. All alloys exhibited excellent tensile ductility at room temperature, in agreement with bend ductility measurements.

TABLE 5. BEND DUCTILITIES OF DISPERSION-EFFECTIVENESS
ALLOYS AT 25 AND -195 C (75 AND -320 F)^(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Minimum Bend Radius Value, T ^(b) , at Indicated Temperature | |
|---|---------------------|--|-----------------|
| | | 25 C (75 F) | -195 C (-320 F) |
| 5W-2.5Mo | 268G | 0, 0 | 0, 0 |
| 5W-2.5Mo-0.07C | 311A | 0, 0 | 0, >18 |
| 5W-2.5Mo-1Hf | 319 | 0, 0 | 0, 2 |
| 5W-2.5Mo-2Hf | 320 | 0, 0 | 5, 5 |
| 5W-2.5Mo-0.5Zr | 313 | 0, 0 | 0, 1 |
| 5W-2.5Mo-1Zr | 314 | 0, 1 | 2, 9 |
| 5W-2.5Mo-1Hf-0.07C | 321B ^(c) | 0, 0 | 2, 17 |
| 5W-2.5Mo-0.5Zr-0.07C | 315O ^(c) | 0 | 1 |
| 5W-2.5Mo-0.5Zr-0.07C | 315P ^(c) | 0 | 2 |
| 5W-2.5Mo-1Zr-0.07C | 317C ^(c) | 0, 0 | 4, 5 |

(a) Recrystallized material.

(b) T-value is radius of last good die before evidence of cracking appears, divided by specimen thickness.

(c) Fabricated according to revised schedule; no 2205 C (4000 F) process anneal.

TABLE 6. ROOM-TEMPERATURE TENSILE PROPERTIES OF
TANTALUM-BASE ALLOYS(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Ultimate Tensile Strength, 1000 psi | Yield Strength, 0.2 Per Cent Offset, 1000 psi | Elongation in 1/2 Inch, per cent | Minimum Bend Radius Value, T(b) |
|---|-------------------|--|--|--|---------------------------------------|
| 5W-2.5Mo | (C) | 111.0 | 104.2 | 16(d) | 0 |
| 5W-2.5Mo-1Hf-0.07C | 321A | 112.0 | 88.3 | 28 | 0(e) |
| 5W-2.5Mo-0.5Zr-0.07C | 315P | 115.0 | 87.3 | 25 | 0 |
| 5W-2.5Mo-0.5Zr-0.07C | 315Q | 113.0 | 94.2 | 30 | -- |
| 5W-2.5Mo-1Zr-0.07C | 317B | 123.0 | 96.7 | 22 | 0(f) |

(a) Recrystallized material. No solution anneal was used in fabrication. Tested using conventional hydraulic loading and a crosshead speed of 0.025 inch per minute for the entire test to fracture (corresponding to an approximate strain rate of 0.05 inch per inch per minute, for a 1/2-inch reduced section).

(b) T-value is radius of last good die before evidence of cracking appears, divided by specimen thickness.

(c) Data from Reference 3. Partially wrought material. Crosshead speed of 0.02 inch per minute up to the point of yielding, and 0.05 inch per minute to fracture (corresponding to an approximate strain rate of 0.016 and 0.04 inch per inch per minute, respectively, for a 1-1/4-inch reduced section).

(d) Elongation in 1 inch.

(e) Value for Specimen 321B.

(f) Value for Specimen 317C.

Table 7 gives tensile data for the Ta-5W-2.5Mo base and the Ta-5W-2.5Mo-0.5Zr-0.07C alloy at 1480 and 1650 C (2700 and 3000 F), and for all dispersion-effectiveness alloys at 1925 C (3500 F). At 1480 C (2700 F), the strength of the base composition decreases monotonically as the structure degenerates from the fibered to the recrystallized condition. In the fibered condition, the carbide dispersion adds little to the alloy's strength. However, the carbide-containing alloy appears relatively insensitive to the strengthening effects of cold working. Thus, in the fibered condition, the "ZrC" dispersion is not particularly effective, but in the recrystallized condition, it is. Tests on duplicate material (Heat No. 315N) showed excellent strength duplication.

At 1650 C (3000 F), comparison of the effect of the "ZrC" dispersion is not rigorous owing to structural differences in the comparative materials. However, it does appear that the dispersion alloy retains about the same strength advantage over the base composition at 1650 C (3000 F) as it enjoyed at 1480 C (2700 F) in the recrystallized condition. Strength duplication (yield strength) of the carbide-containing alloy was poor, and tempers the observation regarding dispersion effectiveness.

At 1925 C (3500 F), the specimens represented various fabrication histories as explained previously. Even considering the dubious effect of the structure (partially fibered) of the Ta-5W-2.5Mo base, it is quite obvious from the data that simple reactive metal (Zr, Hf) additions are markedly strengthening, and increase tensile strengths (both yield and ultimate) by at least 50 per cent at the 1 to 2 per cent addition level. By comparison, carbon additions are ineffective. Furthermore, carbon additions to reactive-metal-containing alloys result in decreased strengths, regardless of prior fabrication history (the Ta-5W-2.5Mo-1Hf-0.07C alloy received the solution process anneal, without benefit to hot strength). The abnormally high elongation values observed for alloys containing both reactive metal and carbon additions where processing did not include a solution anneal may indicate structural instability at the test temperature. Duplicate test results for the Ta-5W-2.5Mo-0.5Zr-0.07C alloy (315Q) at 1925 C (3500 F) showed only fair agreement between samples.

Stress-Rupture Evaluation. Stress-rupture data for recrystallized tantalum, Ta-5W-2.5Mo, and Ta-5W-2.5Mo-0.5Zr-0.07C alloys at 1480 and 1925 C (2700 and 3500 F) are presented in Table 8 and illustrated in Figures 6 and 7, respectively. These data show that a significant increase in rupture parameters is effected at 1480 C (2700 F) when 0.5Zr plus 0.07C is added to a Ta-5W-2.5Mo base. At 1925 C (3500 F) the zirconium-carbon addition is modestly effective for short-time applications. However, as the time variable increases, the dispersion becomes much less effective until at a time somewhat beyond 10 hours, no superiority for the dispersed carbide phase is observed.

The estimated 1- and 10-hour rupture stresses have thus been established and are given in Table 9. These values demonstrate the very desirable behavior of the "ZrC" dispersion at 1480 C (2700 F), and its limited effectiveness at 1925 C (3500 F). Response of the more attractive Ta-5W-2.5Mo-(Zr, Hf) alloys to the time variable was not evaluated.

TABLE 7. TENSILE PROPERTIES OF DISPERSION-EFFECTIVENESS ALLOYS AT 1480 TO 1925 C (2700 TO 3500 F)^(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Condition (b) | Structure (c) | Ultimate Tensile Strength, 1000 psi | Yield Strength, 0.2 Per Cent Offset, 1000 psi | Elongation, per cent in 1/2 in. |
|---|----------------------|--|---------------|--|--|---------------------------------------|
| <u>1480 C (2700 F)</u> | | | | | | |
| 5W-2.5Mo | (d) 268G 268C | WR 65% at 370 C/1 Hr 1300 C/CR 55%/1 Hr 1400 C PR 65% at 1650 C/1 Hr 2205 C/WR 65% at 425 C/1 Hr 1205 C PR 65% at 1650 C/1 Hr 2205 C/WR 65% at 425 C/1 Hr 1650 C | PW W R | 30.3 34.5 23.3 | 18.8 31.8 18.6 | -- 14 34 |
| 5W-2.5Mo-0.5Zr-0.07C | 315N 315N 315N | PR 65% at 1650 C/1 Hr 1650 C/WR 50% at 425 C/1 Hr 1205 C PR 65% at 1650 C/1 Hr 1650 C/WR 50% at 425 C/1 Hr 1760 C PR 65% at 1650 C/1 Hr 1650 C/WR 50% at 425 C/1 Hr 1760 C | W R R | 35.2 38.0 38.6 | 25.2 32.5 (e) | 48 56 30 |
| <u>1650 C (3000 F)</u> | | | | | | |
| 5W-2.5Mo | (d) | WR 65% at 370 C/1 Hr 1300 C/CR 55%/1 Hr 1400 C | PW | 17.0 | 12.1 | -- 66 |
| 5W-2.5Mo-0.5Zr-0.07C | 315P 315P | PR 65% at 1650 C/1 Hr 1650 C/WR 50% at 425 C/1 Hr 1760 C PR 65% at 1650 C/1 Hr 1650 C/WR 50% at 425 C/1 Hr 1760 C | R R | 23.2 (f) | 19.0 14.6 | -- 76 -- >13 |
| <u>1925 C (3500 F)</u> | | | | | | |
| 5W-2.5Mo | (d) | WR 65% at 370 C/1 Hr 1300 C/CR 55%/1 Hr 1400 C | PW | 9.0 | 8.0 | -- 90 |
| 5W-2.5Mo-0.07C | 311A | PR 65% at 1650 C/1 Hr 2205 C/WR 60% at 480 C/1 Hr 1760 C | R | 10.6 | 10.4 | -- 73 |
| 5W-2.5Mo-0.13C | 312A | PR 65% at 1650 C/1 Hr 2205 C/1 Hr 1760 C | R | 10.2 | 8.6 | -- 54 |
| 5W-2.5Mo-1Hf | 319 | PR 65% at 1650 C/1 Hr 2205 C/PR 65% at 980 C/1 Hr 1760 C | R | 14.1 | 14.1 | -- 47 |
| 5W-2.5Mo-2Hf | 320 | PR 65% at 1650 C/1 Hr 2205 C/PR 65% at 980 C/1 Hr 1760 C | R | 13.7 | 13.7 | -- 54 |
| 5W-2.5Mo-0.5Zr | 313 | PR 65% at 1650 C/1 Hr 2205 C/WR 65% at 425 C/1 Hr 1760 C | R | 12.3 | 11.8 | -- 50 |
| 5W-2.5Mo-1Zr | 314 | PR 65% at 1650 C/1 Hr 2205 C/WR 60% at 480 C/1 Hr 1760 C | R | 15.1 | 13.8 | -- 32 |
| 5W-2.5Mo-1Hf-0.07C | 321 | PR 65% at 1650 C/1 Hr 2205 C/PR 50% at 980 C/1 Hr 1760 C | R | 11.2 | 10.1 | 4 -- |
| 5W-2.5Mo-0.5Zr-0.07C | 315Q 315Q | PR 65% at 1650 C/1 Hr 1650 C/WR 50% at 425 C/1 Hr 1760 C PR 65% at 1650 C/1 Hr 1650 C/WR 50% at 425 C/1 Hr 1760 C | R R | 10.9 12.2 | 9.8 11.8 | 130 -- 148 -- |
| 5W-2.5Mo-0.5Zr-0.13C | 318A | PR 75% at 1650 C/1 Hr 1760 C | R | 10.1 | 8.5 | -- >24(h) |
| 5W-2.5Mo-1Zr-0.07C | 317B | PR 75% at 1650 C/1 Hr 1760 C | R | 12.1 | 11.8 | 130 -- |
| 5W-2.5Mo-1Zr-0.13C | 316B | PR 75% at 1650 C/1 Hr 1760 C | R | 6.7 | 5.4 | 90 -- |

(a) Tested in vacuum using a mechanical screw-driven crosshead with a speed of 0.02 or 0.05 inch per minute for a 1/2- and 1-1/4-inch reduced section, respectively (corresponding to an approximate strain rate of 0.04 inch per inch per minute).

(b) CR = cold rolled, WR = warm rolled, and PR = pack rolled.

(c) W = wrought, PW = partially wrought, and R = recrystallized.

(d) Data from Reference 3. Crosshead speed of 0.01 inch per minute up to the point of yielding and 0.05 inch per minute to fracture (corresponding to approximate strain rates of 0.008 and 0.04 inch per inch per minute, respectively, for a 1-1/4-inch reduced section).

(e) Short in heater element. Adjusted and test completed. Yield could not be determined.

(f) Element failure after yield point. Tensile strength not determined.

(g) Final rolled 30 per cent at 480 C (900 F) followed by 30 per cent at 980 C (1800 F).

(h) After maximum load, element developed a short and test was discontinued.

TABLE 8. STRESS-RUPTURE PROPERTIES OF TANTALUM, Ta-5W-2.5Mo, AND Ta-5W-2.5Mo-0.5Zr-0.07C AT 1480 AND 1925 C (2700 AND 3500 F)^(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Stress, 1000 psi | Deformation Properties | | Rupture Time, hours | Gage Length, inches | Elongation in Gage Length, per cent |
|---|-------------------|---------------------|---------------------------------------|---|---------------------------|---------------------------|---|
| | | | Elongation on Loading, per cent | Minimum Creep Rate, per cent/hour | | | |
| <u>1480 C (2700 F)</u> | | | | | | | |
| 100Ta | (b) | 5.26 | -- | -- | 0.4 ^(c) | 1.20 | 54 |
| | (b) | 4.75 | 2.02 | -- | 0.37 | 1.20 | 42 |
| | (b) | 4.2 | 0.80 | 4.8 | 3.3 ^(d) | 1.20 | 68 |
| | (b) | 3.9 | 0.84 | 2.5 | 7.6 | 1.20 | 74 |
| 5W-2.5Mo | (b) | 25.0 | 9.48 | -- | 0.023 | 1.25 | 33 |
| | (b) | 15.0 | 1.05 | 5.44 | 2.7 | 1.25 | 37 |
| | 268C-1 | 13.0 | 0.95 | 4.75 | 4.1 | 1.00 | 35 |
| 5W-2.5Mo-0.5Zr-0.07C | 315L-1 | 17.0 | 0.02 | 0.582 | 20.85 | 0.50 | 44 |
| | 315L-2 | 23.0 | 0.06 | 2.42 | 3.72 | 0.50 | 64 |
| | 315L-3 | 28.0 | 0.1 | 11.1 | 1.13 | 0.50 | 56 |
| <u>1925 C (3500 F)</u> | | | | | | | |
| 100Ta | 191-5 | 1.5 | 0.48 | 0.679 | 7.5 ^(e) | 1.00 | 49 |
| | 191-6 | 2.2 | -- | -- | 0.034 | 1.00 | 53 |
| | 191-7 | 1.8 | 3.65 | -- | 0.05 | 1.00 | 62 |
| | 191-8 | 1.6 | 2.42 | -- | 0.092 | 1.00 | 43 |
| 5W-2.5Mo | 268C-2 | 6.2 | 0.23 | 162.5 | 0.5 | 1.00 | 86 |
| | 268C-3 | 3.5 | 0.015 | 2.55 | 12.2 | 1.00 | 98 |
| | 268C-4 | 4.5 | 0 | 15.59 | 2.63 | 1.00 | 79 |
| 5W-2.5Mo-0.5Zr-0.07C | 315M-1 | 8.0 | 0.03 | 103.0 | 0.5 | 0.50 | 134 |
| | 315M-2 | 5.5 | 0 | 58.75 | 1.55 ^(f) | 0.50 | 100 |
| | 315M-3 | 3.0 | 0 | 2.65 | >15.0 ^(g) | 0.50 | 48 |

(a) Recrystallized material.

(b) Data from Reference 3.

(c) Tested at 1440 C (2620 F).

(d) Tantalum heater element failure after 23 minutes; element replaced and specimen reloaded. Rupture time is the total for both tests.

(e) Hardness values after test indicate specimen contaminated during test.

(f) Small inclusion in reduced section.

(g) Test discontinued after 15 hours. Specimen in latter portion of second-stage creep. Rupture estimated from creep rates to be 30 hours.

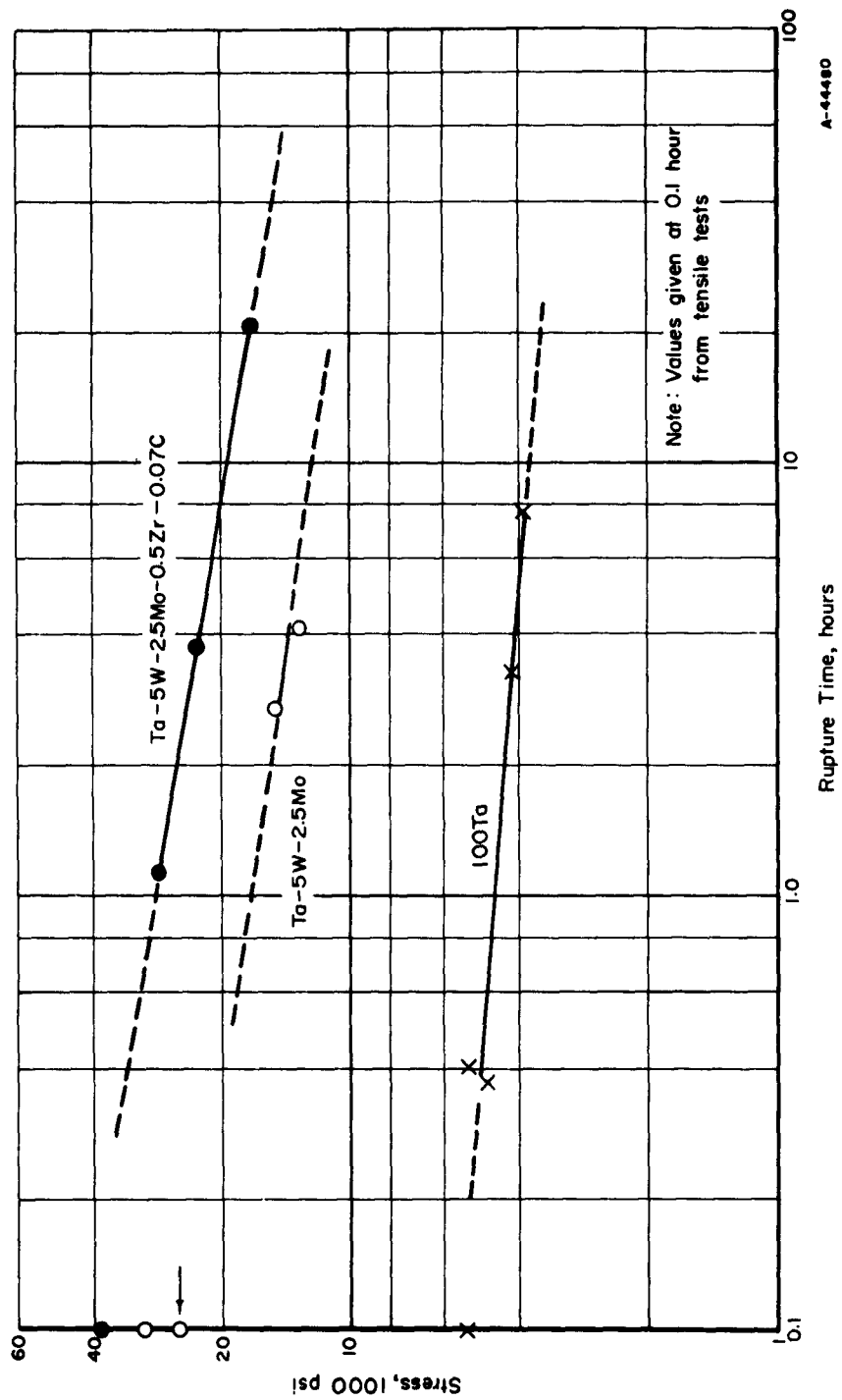


FIGURE 6. STRESS-RUPTURE CURVES FOR RECRYSTALLIZED TANTALUM, Ta-5W-2.5Mo, AND Ta-5W-2.5Mo-0.5Zr-0.07C AT 1480 C (2700 F)

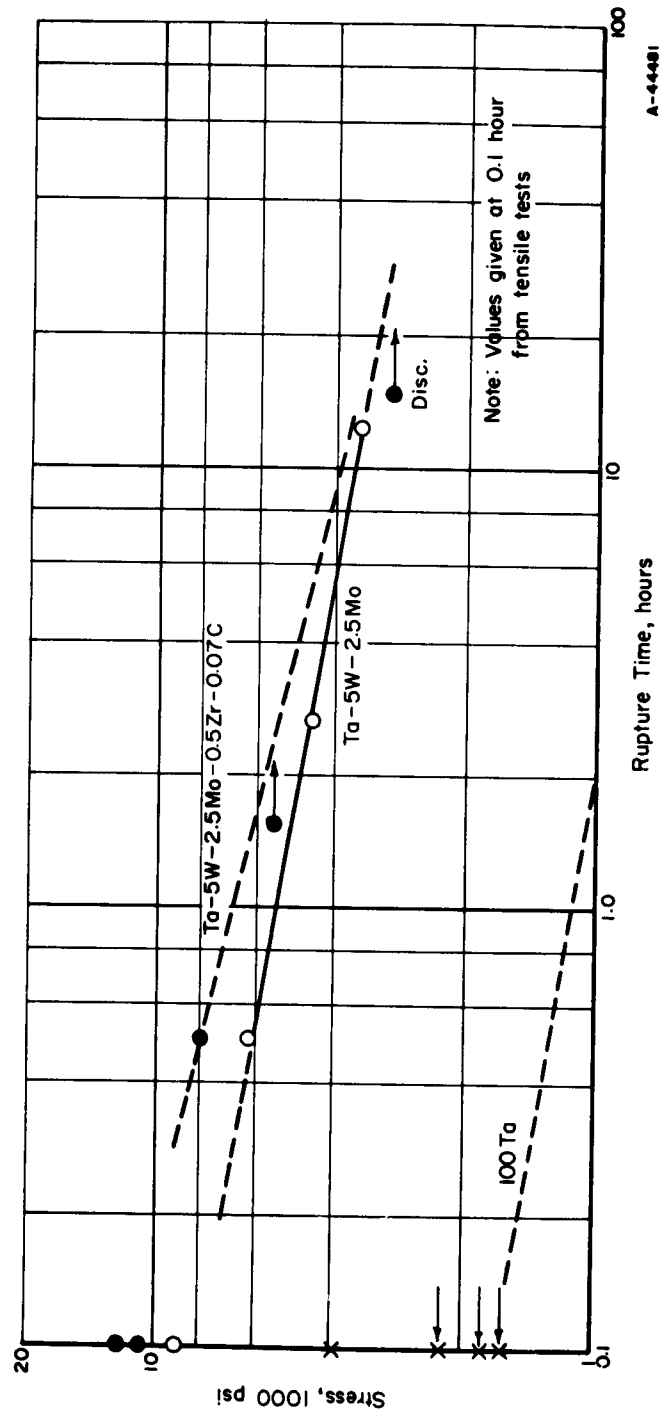


FIGURE 7. STRESS-RUPTURE CURVES FOR RECRYSTALLIZED TANTALUM, Ta-5W-2.5Mo, AND Ta-5W-2.5Mo-0.5Zr-0.07C AT 1925 C (3500 F)

TABLE 9. RUPTURE STRENGTHS OF TANTALUM, Ta-5W-2.5Mo,
AND Ta-5W-2.5Mo-0.5Zr-0.07C ALLOYS AT 1480
AND 1925 C (2700 AND 3500 F)(a)

| Alloy Composition (Balance Tantalum), weight per cent | Ultimate Tensile Strength, 1000 psi | Stress to Rupture ^(b) , 1000 psi, at Indicated Time | |
|---|---|--|---------|
| | | 1 Hour | 10 Hour |
| <u>1480 C (2700 F)</u> | | | |
| 100Ta ^(c) | 5.3 | 4.6 | 3.8 |
| 5W-2.5Mo | 30.3 ^(c) | 17.0 | 12.1 |
| 5W-2.5Mo-0.5Zr-0.07C | 38.3 ^(d) | 28.5 | 19.2 |
| <u>1925 C (3500 F)</u> | | | |
| 100Ta | 3.9 ^(c) | 1.12 | 0.75 |
| 5W-2.5Mo | 9.0 ^(c) | 5.35 | 3.6 |
| 5W-2.5Mo-0.5Zr-0.07C | 11.6 ^(d) | 6.7 | 3.9 |

(a) Recrystallized material.

(b) Rupture strengths obtained graphically from a stress-rupture plot.

(c) Data from Reference 3.

(d) Average of two tests.

Recrystallization Behavior. The recrystallization temperature for several solid solution plus dispersion strengthened tantalum-base alloys are given in Table 10, based on the results of microstructural examination and hardness measurements presented in Table 11. These alloys showed relatively high recrystallization temperatures [1600 to 1800 C (2910 to 3270 F)] but no consistent improvement could be attributed to the dispersed phases.

Welding. Automatic TIG welding for solid solution plus carbide dispersion strengthened tantalum-base alloys was done in an inert-gas-filled chamber. The chamber was pumped down to less than 1 micron prior to filling with helium. Welding conditions were

| | |
|--------------|------------------|
| Arc voltage | 16 to 17 volts |
| Arc current | 76 to 96 amperes |
| Travel speed | 7 ipm. |

Details of the automatic TIG welding conditions for specific alloys are given in Appendix II.

Results of visual and radiographic examination of automatic TIG welds for the dispersion-effectiveness alloys are given in Table 12. Welds of the Ta-5W-2.5Mo-base dispersion alloys exhibited problems in weld mismatch and discoloration of weld metal that were not observed so extensively for the base composition. Specimens 317B and 316B cracked on welding, which further suggests welding problems associated with a dispersion addition of 1 per cent zirconium and 0.07 to 0.13 per cent carbon. Carbon-free dispersion-effectiveness alloys were not evaluated.

Weld-ductility results for these carbide dispersion-effectiveness alloys are given in Table 13. All solid solution plus carbide dispersion strengthened alloys were brittle in the as-welded condition. However, after a simulated service thermal exposure of 1 hour at 1925 C (3500 F) significant recovery of weld ductility at room temperature was observed. Only the two high-carbon (0.13 per cent)-containing alloys showed no ductility improvement following the high-temperature exposure.

Effects of Fabrication on a Dispersion-Containing Alloy

For this phase of the investigation the Ta-5W-2.5Mo-0.5Zr-0.07C alloy was selected to study the effects of processing variables on the mechanical and metallurgical behavior of a dispersion-containing alloy.

Fabrication

Details of the fabrication schedule selected for seven button ingots of the Ta-5W-2.5Mo-0.5Zr-0.07C alloy are illustrated in Figure 8. Ingots 315B, C, and D were used to investigate the effects of intermediate annealing at 1650, 1925, and 2205 C (3000, 3500, and 4000 F) coupled with rolling 50 per cent at 425 and 980 C (800 and 1800 F).

TABLE 10. RECRYSTALLIZATION TEMPERATURES FOR DISPERSION-EFFECTIVENESS ALLOYS

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Condition(a) | Recrystallization Temperature(b) | |
|---|-------------------|--|-------------------------------------|------|
| | | | C | F |
| 5W-2.5Mo | 268G | PR 65% at 1650 C/1 hr 2205 C/WR 65% at 425 C | 1700 | 3090 |
| 5W-2.5Mo-0.07C | 311A | PR 65% at 1650 C/1 hr 2205 C/WR 60% at 480 C | 1700 | 3090 |
| 5W-2.5Mo-0.13C | 312A | PR 65% at 1650 C/1 hr 2205 C/(c) | 1700 | 3090 |
| 5W-2.5Mo-1Hf | 319 | PR 65% at 1650 C/1 hr 2205 C/PR 65% at 980 C | 1700 | 3090 |
| 5W-2.5Mo-2Hf | 320 | PR 65% at 1650 C/1 hr 2205 C/PR 65% at 980 C | 1600 | 2910 |
| 5W-2.5Mo-0.5Zr | 313 | PR 65% at 1650 C/1 hr 2205 C/WR 65% at 425 C | 1700 | 3090 |
| 5W-2.5Mo-1Zr | 314 | PR 65% at 1650 C/1 hr 2204 C/WR 60% at 480 C | 1700 | 3090 |
| 5W-2.5Mo-1Hf-0.07C | 321 | PR 65% at 1650 C/1 hr 2205 C/PR 50% at 980 C | 1800 | 3270 |

(a) WR = warm rolled

PR = pack rolled.

(b) Complete recrystallization (75 per cent or greater) after 1-hour exposure in vacuum.

(c) Final rolled 30 per cent at 480 C (900 F) followed by 30 per cent at 980 C (1800 F).

TABLE 11. MICROSTRUCTURES AND HARDNESSES OF DISPERSION-EFFECTIVENESS ALLOYS^(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Cast, RT | Wrought, RT | Microstructure ^(b) and Hardness ^(c) , VHN, After Annealing 1 Hour at Indicated Temperature | | | | | | | | | |
|---|-------------------|-------------|----------------|--|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--|
| | | | | 1100 C (2010 F) | 1200 C (2190 F) | 1300 C (2370 F) | 1400 C (2550 F) | 1500 C (2730 F) | 1600 C (2910 F) | 1700 C (3090 F) | 1800 C (3270 F) | 1900 C (3450 F) | |
| 5W-2.5Mo | 268G | -- | W 213 | W 281 | W 270 | W 272 | Rp 243 | Rp 256 | Rp 227 | R 225 | R 253 | R 312 | |
| 5W-2.5Mo-0.07C | 311A | -- | W 272 | W 319 | W 314 | W 304 | W 281 | Rp 276 | Rp 236 | R 240 | R 242 | R 264 | |
| 5W-2.5Mo-0.13C | 312A | -- | W 309 | W 312 | W 302 | W 297 | W 279 | W 256 | W 245 | R 243 | R 245 | R 270 | |
| 5W-2.5Mo-1Hf | 319 | -- | W 228 | W 312 | W 294 | W 281 | Rp 266 | Rp 274 | Rp 270 | R 276 | R 260 | R 262 | |
| 5W-2.5Mo-2Hf | 320 | -- | W 242 | W 345 | W 325 | W 302 | Rb 292 | Rp 266 | R 297 | R 289 | R 279 | R 314 | |
| 5W-2.5Mo-0.5Zr | 313 | -- | W 222 | W 339 | W 306 | W 292 | Rp 274 | Rp 283 | Rp 276 | R 264 | R 260 | R 256 | |
| 5W-2.5Mo-1Zr | 314 | -- | W 233 | W 376 | W 306 | W 287 | Rb 276 | Rp 256 | Rp 268 | R 289 | R 292 | R 289 | |
| 5W-2.5Mo-1Hf-0.07C | 321 | -- | W 289 | W 339 | W 297 | W 314 | Rb 274 | Rp 283 | Rp 260 | Rp 266 | R 266 | R 272 | |

(a) Wrought condition given in Table 10.

(b) W = wrought

Rb = recrystallization beginning

Rp = recrystallization partially complete

R = recrystallization essentially complete

Minimum temperature for complete recrystallization (75 per cent or greater) is underlined.

(c) Hardness values are the average of five impressions using a 10-kg load.

TABLE 12. RESULTS OF EXAMINATION OF AUTOMATIC TIG-PRODUCED
WELDS FOR DISPERSION-EFFECTIVENESS ALLOYS

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Visual | Radiographic |
|---|-------------------|---|---------------------|
| 5W-2.5Mo | 268G | Slightly mismatched at one end | No defects observed |
| 5W-2.5Mo-1Hf-0.07C | 321A | Mismatched, weld discolored | Ditto |
| 5W-2.5Mo-0.5Zr-0.07C | 315N | Mismatched at start end, nonuniform penetration, weld discolored | " |
| 5W-2.5Mo-0.5Zr-0.13C | 318A | Mismatched, weld discolored | " |
| 5W-2.5Mo-1Zr-0.07C | 317B | Mismatched, 1/2-inch- long crack at start, weld discolored | Confirmed crack |
| 5W-2.5Mo-1Zr-0.13C | 316B | Excessive penetration, crack at start end, weld discolored | Ditto |

TABLE 13. AUTOMATIC TIG WELD-BEND DUCTILITIES OF DISPERSION-EFFECTIVENESS ALLOYS^(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Condition | Minimum Bend Radius Value, T ^(b) , at Room Temperature |
|---|-------------------|---------------------------------------|---|
| 5W-2.5Mo | 268G | Base | 0, 0 |
| | 268G | As welded | 2, 2 |
| | 268G | As welded + 1 hour 1925 C (3500 F) | 0 |
| 5W-2.5Mo-1Hf-0.07C | 321B | Base | 0, 0 |
| | 321A | As welded | >25, >26 |
| | 321A | As welded + 1 hour 1925 C (3500 F) | 4 |
| 5W-2.5Mo-0.5Zr-0.07C | 315O | Base | 0 |
| | 315P | Base | 0 |
| | 315N | As welded | 24, 50 |
| | 315N | As welded + 1 hour 1925 C (3500 F) | 12 |
| 5W-2.5Mo-0.5Zr-0.13C | -- | Base | -- |
| | 318A | As welded | >24, >24 |
| | 318A | As welded + 1 hour 1925 C (3500 F) | 24 |
| 5W-2.5Mo-1Zr-0.07C | 317C | Base | 0, 0 |
| | 317B | As welded | >25, >25 |
| | 317B | As welded + 1 hour 1925 C (3500 F) | 16 |
| 5W-2.5Mo-1Zr-0.13C | -- | Base | -- |
| | 316B | As welded | 24 |
| | 316B | As welded + 1 hour 1925 C (3500 F) | 24 |

(a) Recrystallized material.

(b) T-value is radius of last good die before evidence of cracking appears divided by specimen thickness.

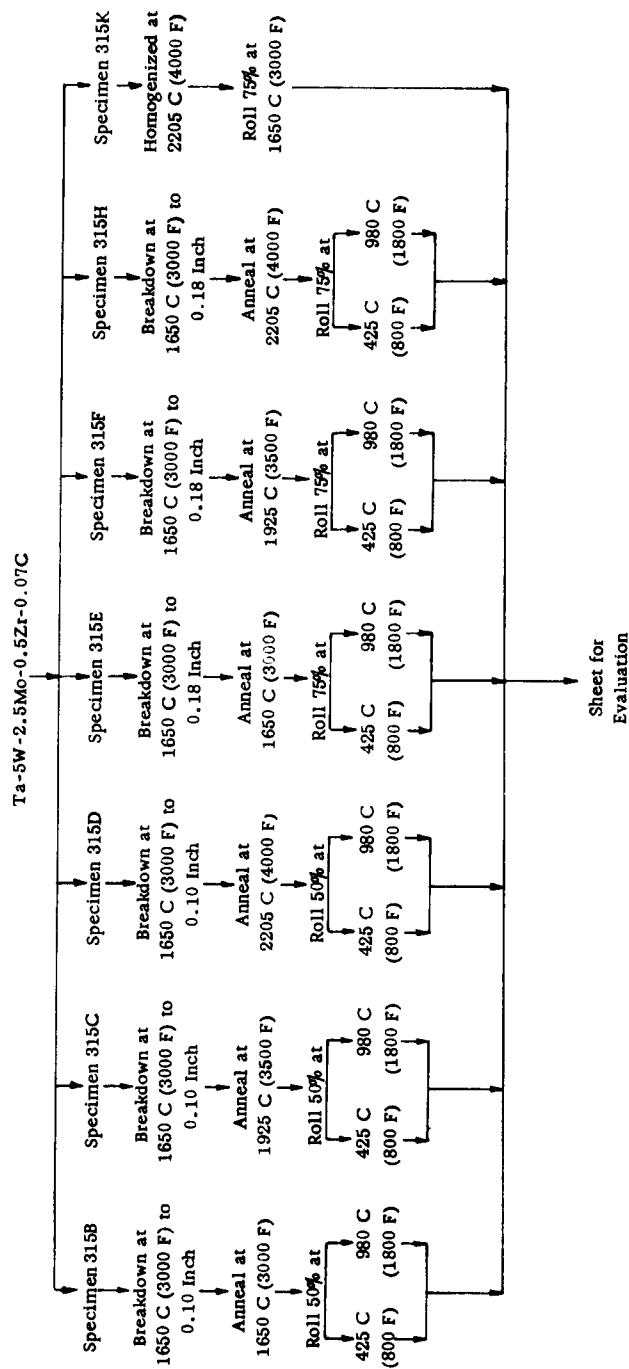


FIGURE 8. FABRICATION SCHEDULE FOR THE Ta-5W-2.5Mo-0.5Zr-0.07C ALLOY

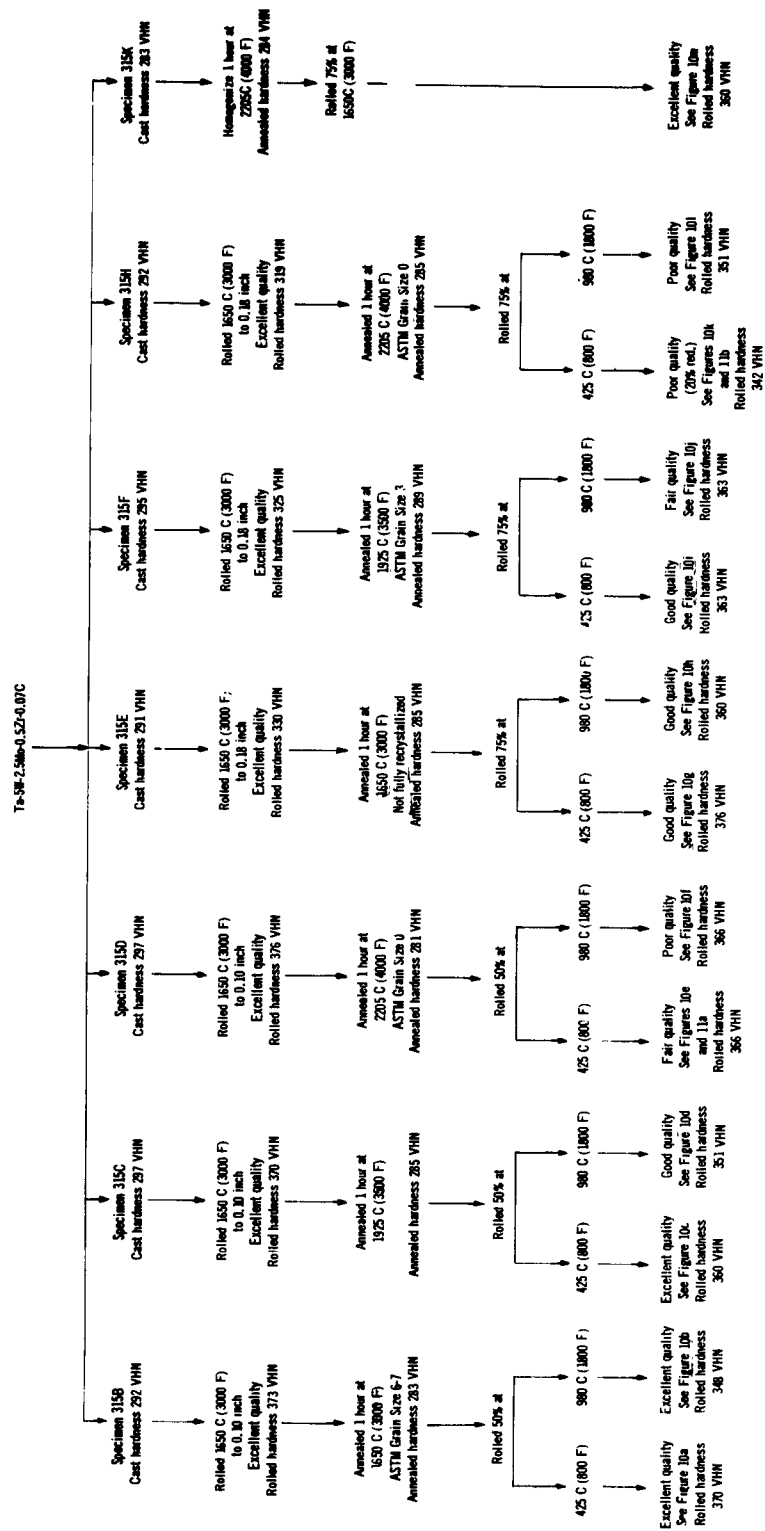
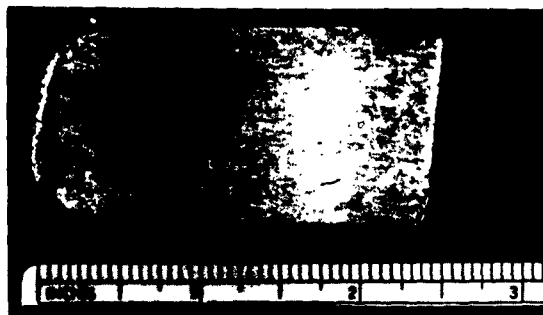


FIGURE 9. FABRICATION FLOW CHART FOR THE Ta-5W-2.5Mo-0.5Zr-0.07C ALLOY

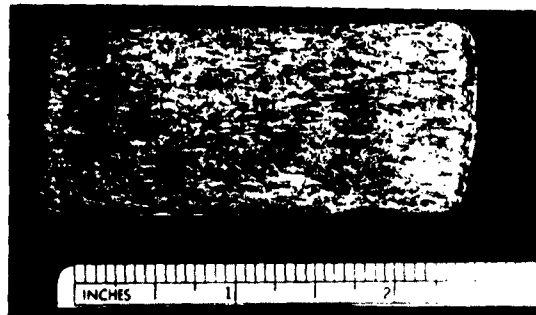


1X

a. Specimen 315B-L

N92874

Annealed 1 hour at 1650 C (3000 F); rolled 50% at 425 C (800 F); excellent quality, 370 VHN.

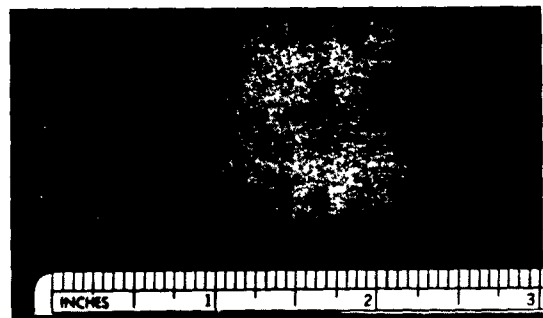


1X

b. Specimen 315B-H

N93048

Annealed 1 hour at 1650 C (3000 F); rolled 50% at 980 C (1800 F); excellent quality, 348 VHN.

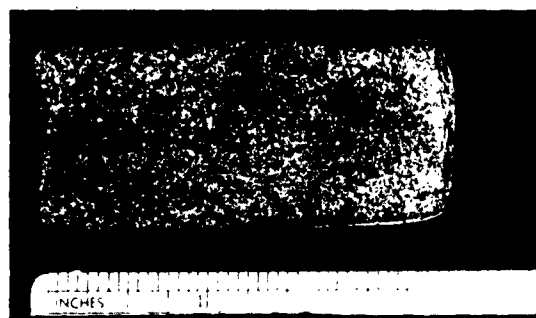


1X

c. Specimen 315C-L

N92875

Annealed 1 hour at 1925 C (3500 F); rolled 50% at 425 C (800 F); excellent quality, 360 VHN.

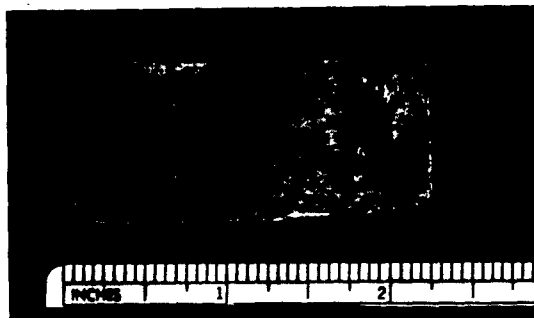


1X

d. Specimen 315C-H

N93049

Annealed 1 hour at 1925 C (3500 F); rolled 50% at 980 C (1800 F); good quality, 351 VHN.



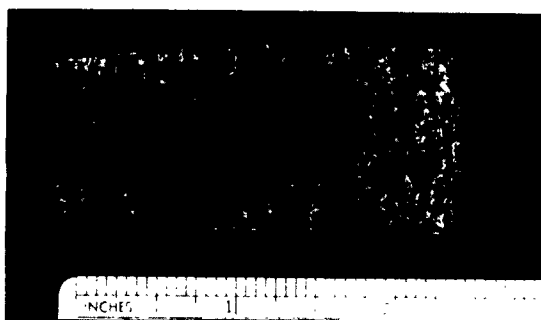
1X

e. Specimen 315D-L

N92876

Annealed 1 hour at 2205 C (4000 F); rolled 50% at 425 C (800 F); fair quality, 366 VHN.

FIGURE 10. EFFECT OF ANNEALING TEMPERATURE, REDUCTION, AND ROLLING TEMPERATURE ON FINAL SHEET QUALITY OF THE Ta-5W-2.5Mo-0.5Zr-0.07C ALLOY



1X

f. Specimen 315D-H

N93050

Annealed 1 hour at 2205 C (4000 F); rolled 50% at 980 C (1800 F); poor quality, 366 VHN.

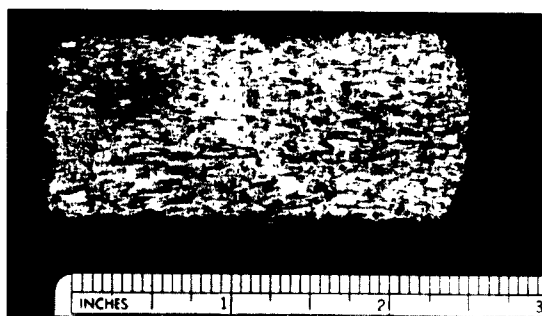


1X

g. Specimen 315E-L

N92877

Annealed 1 hour at 1650 C (3000 F); rolled 75% at 425 C (800 F); good quality, 376 VHN.

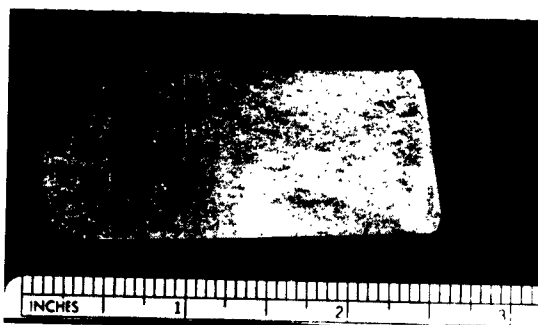


1X

h. Specimen 315E-H

N93051

Annealed 1 hour at 1650 C (3000 F); rolled 75% at 980 C (1800 F); good quality, 360 VHN.



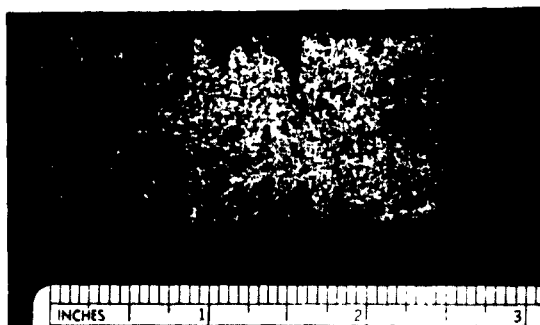
1X

i. Specimen 315F-L

N92878

Annealed 1 hour at 1925 C (3500 F); rolled 75% at 425 C (800 F); good quality, 363 VHN.

FIGURE 10. (CONTINUED)

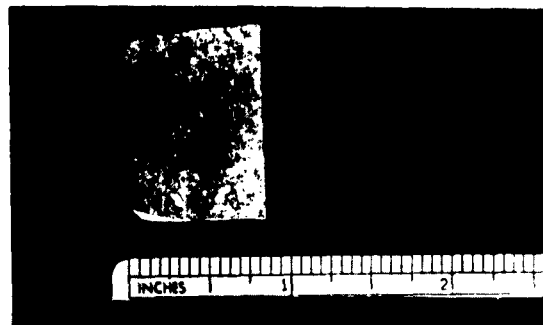


1X

j. Specimen 315F-H

N93052

Annealed 1 hour at 1925 C (3500 F); rolled 75% at 980 C (1800 F); fair quality, 363 VHN.



1X

k. Specimen 315H-L

N92879

Annealed 1 hour at 2205 C (4000 F); rolled 20% at 425 C (800 F); poor quality, 342 VHN.



1X

l. Specimen 315H-H

N93053

Annealed 1 hour at 2205 C (4000 F); rolled 75% at 980 C (1800 F); poor quality, 351 VHN.



1X

m. Specimen 315K

N92880

Annealed 1 hour at 2205 C (4000 F); rolled 75% at 1650 C (3000 F); excellent quality, 360 VHN.

FIGURE 10. (CONTINUED)

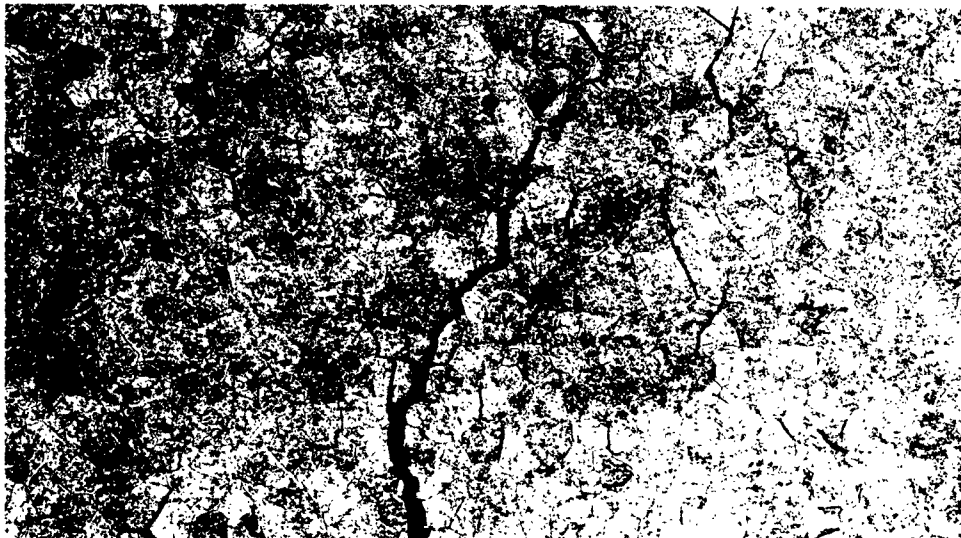


10X

N92881

a. Specimen 315D-L

Annealed 1 hour at 2205 C (4000 F); rolled
50% at 425 C (800 F); 366 VHN.



10X

N92882

b. Specimen 315H-L

Annealed 1 hour at 2205 C (4000 F); rolled
20% at 425 C (800 F); 342 VHN.

FIGURE 11. CLOSE-UP OF AS-ROLLED SURFACE OF THE Ta-5W-2.5Mo-0.5Zr-0.07C
ALLOY SHOWING MODE OF CRACKING

Ingots 315E, F, and H incorporated a 75 per cent final reduction, with other variables the same as for Ingots 315B, C, and D. Ingot 315K, following a homogenization treatment at 2205 C (4000 F), was rolled at 1650 C (3000 F) directly to sheet. Specimens were prepared for mechanical testing in both the stress-relieved and recrystallized conditions.

Table 14 presents fabrication data for the Ta-5W-2.5Mo-0.5Zr-0.07C alloy. All ingots (except Specimen 315K which was rolled directly to sheet) were broken down to excellent 0.100 to 0.180-inch strip at 1650 C (3000 F).

Annealing for 1 hour at 2205 C (4000 F) prior to final sheet rolling greatly impaired fabricability, and only limited material for testing was obtained. A process-annealing temperature of 1650 C (3000 F) was much more desirable for the production of high-quality sheet, as evidenced by the fabrication data presented in Table 14. Detail results of the fabrication study are summarized in the flow chart, Figure 9, and photographs showing final sheet quality, Figures 10 and 11.

The reasons for the drastic effects of high-temperature process annealing upon fabricability were revealed in a metallographic study of the Ta-5W-2.5Mo-0.5Zr-0.07C alloy at various stages of processing.

Figure 12 shows a typical cast microstructure. Figure 13 illustrates the effect of rolling reduction and process-annealing temperature on the structure of the Ta-5W-2.5Mo-0.5Zr-0.07C alloy. The undesirable heavy Widmanstätten precipitation present in alloys annealed at 1925 C (3500 F) and above is not present when the process-annealing treatment is reduced to 1650 C (3000 F). Structure resulting from this lower annealing temperature, finely distributed carbide in a small-grained matrix, allowed

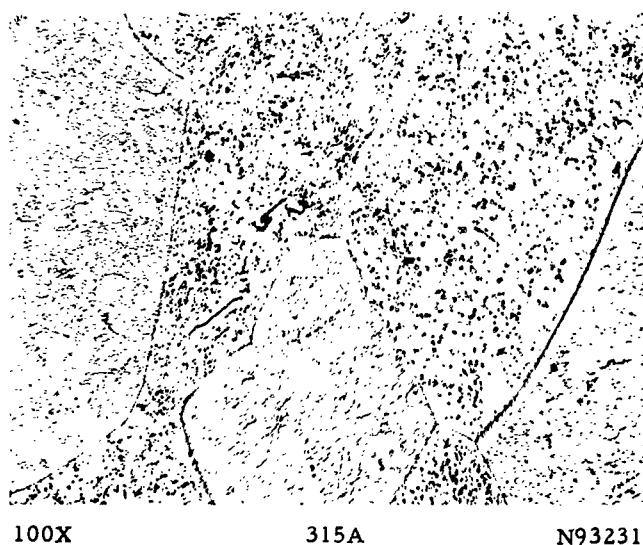


FIGURE 12. TYPICAL MICROSTRUCTURE OF CAST Ta-5W-2.5Mo-0.5Zr-0.07C

TABLE 14. FABRICATION DATA FOR THE Ta-5W-2.5Mo-0.5Zr-0.07C ALLOY

| Alloy Specimen | Cast Hardness(a), VHN | Initial Rolling Temperature(b) | | Quality of Strip(c) | Intermediate Annealing Temperature | | Hardness(a), VHN | | Final Rolling Temperature(b) | | Final Reduction, per cent | Quality of Strip(c) |
|----------------|-----------------------|--------------------------------|------|---------------------|------------------------------------|------|------------------|----------|------------------------------|----------|---------------------------|---------------------|
| | | C | F | | C | F | Rolled | Annealed | C | F | | |
| 315B | 292 | 1650 | 3000 | Excellent | 1650 | 3000 | 373 | 283 | 425/980 | 800/1800 | 50/50 | Excellent/Excellent |
| 315C | 297 | 1650 | 3000 | Excellent | 1925 | 3500 | 370 | 285 | 425/980 | 800/1800 | 50/50 | Excellent/Good |
| 315D | 297 | 1650 | 3000 | Excellent | 2205 | 4000 | 376 | 281 | 425/980 | 800/1800 | 50/50 | Fair/Poor |
| 315E | 291 | 1650 | 3000 | Excellent | 1650 | 3000 | 330 | 285 | 425/980 | 800/1800 | 75/75 | Good(d)/Good |
| 315F | 295 | 1650 | 3000 | Excellent | 1925 | 3500 | 325 | 289 | 425/980 | 800/1800 | 75/75 | Good(e)/Fair |
| 315H | 292 | 1650 | 3000 | Excellent | 2205 | 4000 | 319 | 285 | 425/980 | 800/1800 | 20/75 | Poor/Poor |
| 315K | 283(f) | 1650 | 3000 | -- | -- | -- | -- | -- | -- | -- | 75 | Excellent |

(a) Hardness values are the average of five impressions using a 10-kg load.

(b) Alloys rolled at 425 C (800 F) unprotected in air. Alloys rolled at 980 C (1800 F) in evacuated stainless steel packs. Alloys rolled at 1650 C (3000 F) in evacuated molybdenum packs.

(c) Excellent - no cracking of edges or surface

Good - slight cracking of edges and surface

Fair - considerable cracking of edges and surface

Poor - extensive cracking throughout specimen.

(d) Slight edge cracking; alligator one end. Defects removed by grinding.

(e) Considerable edge cracking; alligator both ends. Defects removed by grinding.

(f) Annealed 1 hour at 2205 C (4000 F) prior to rolling. Hardness after annealing was 284 VHN.

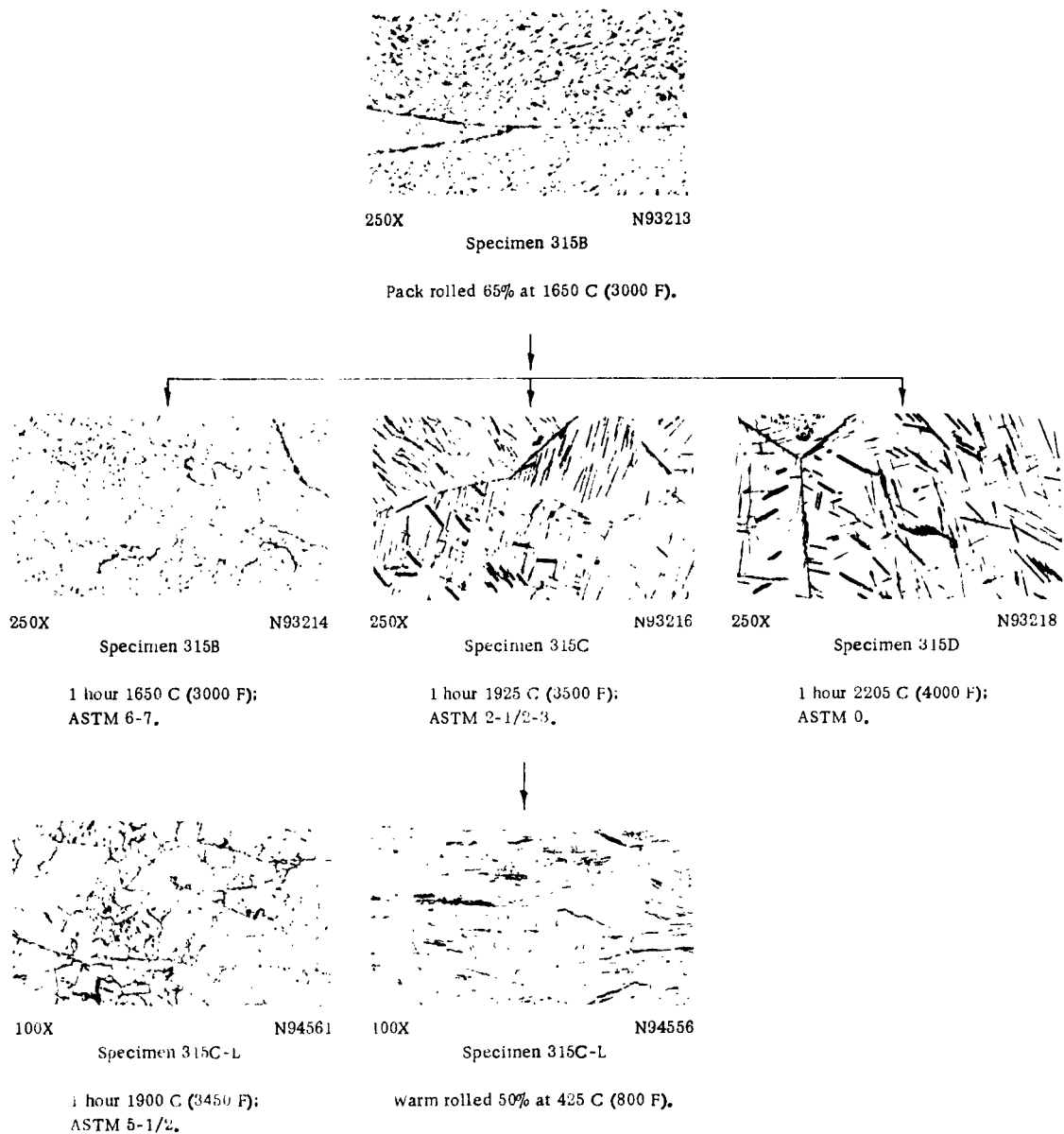


FIGURE 13. EFFECT OF ROLLING AND ANNEALING TEMPERATURE ON THE MICROSTRUCTURE OF THE Ta-5W-2.5Mo-0.5Zr-0.07C ALLOY

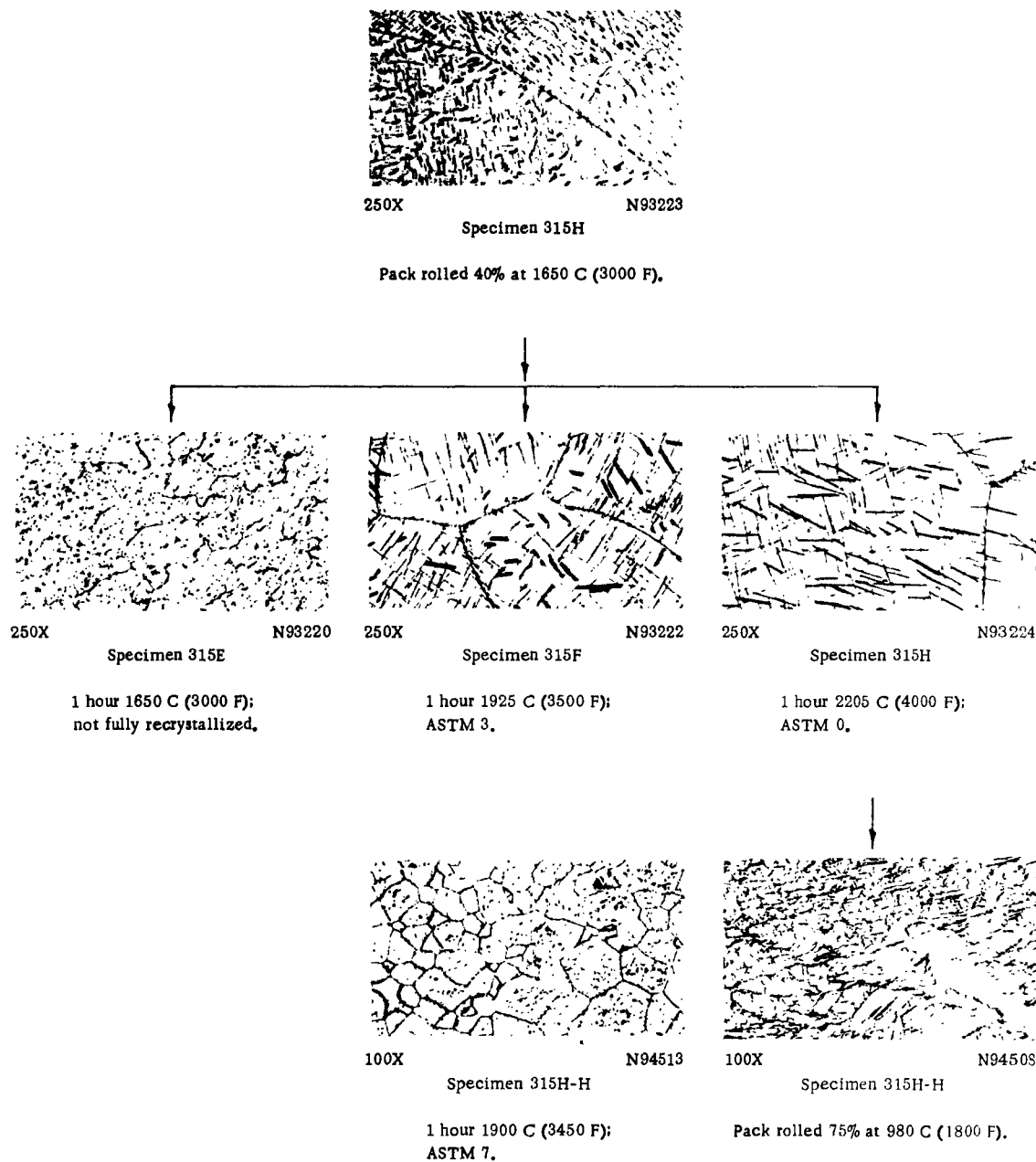


FIGURE 13. (CONTINUED)

consistent final fabrication to high-quality sheet (see Table 14). However, as illustrated in Figure 13, if the large-grained Widmanstätten structure produced by annealing at 1925 C (3500 F) and above is successfully rolled, the grain size produced by annealing at 1700 to 1900 C (3090 to 3450 F) in the final-rolled sheet is relatively fine, regardless of process-annealing temperature and amount of reduction. Material that was recrystallized after rolling from a 1650 C (3000 F) process anneal was somewhat finer grained than material achieved using higher process-annealing temperatures. Note also the disappearance of the Widmanstätten carbide with fabrication and recrystallization annealing at 1900 C (3450 F).

Evaluation

Evaluation of the effects of fabrication history on the Ta-5W-2.5Mo-0.5Zr-0.07C alloy was limited to 1480 C (2700 F) tensile tests and a study of the recrystallization behavior.

Tensile Tests. Tensile data at 1480 C (2700 F) are summarized in Table 15. Varying the initial breakdown rolling from 40 to 65 per cent, annealing at 1650 or 1925 C (3000 or 3500 F), and final rolling 50 to 75 per cent at 425 and 980 C (800 and 1800 F) appeared to have little effect on the tensile properties. Tensile strength ranged from 32,200 to 38,200 psi and yield strength from 22,000 to 28,800 psi.

Specimen 315K, which was process annealed at 2205 C (4000 F), and finish-rolled at 1650 C (3000 F) exhibited significantly greater ultimate strength (41,200 psi) than the materials with more modest thermal treatments incorporated in their process schedule. No superiority was noted in the yield strength value, however. Specimen 315D-L, also process annealed at 2205 C (4000 F), exhibited still higher tensile strength, and a significant improvement in yield strength as well. However, contamination of this specimen during testing introduced a complicating variable, and the validity of the strength values is questionable.

In both cases examined, recrystallization improved both the yield and ultimate strengths of the Ta-5W-2.5Mo-0.5Zr-0.07C alloy at 1480 C (2700 F). The degree of improvement appeared most pronounced for the material with the most severe prior thermal history (Specimen 315K). The 51,200-psi tensile strength measured for this material represents one of the highest strengths attained at 1480 C (2700 F) in tantalum-base alloys to date. This material is about 10 times stronger than unalloyed tantalum and more than twice as strong as Ta-10W at 1480 C (2700 F).

Recrystallization Behavior. Recrystallization temperatures for Ta-5W-2.5Mo-0.5Zr-0.07C fabricated and evaluated in various conditions are given in Table 16, based on data presented in Table 17. Detailed recrystallization performance curves for the fabrication conditions studied are given in Appendix III.

Prior fabrication history is seen to have little effect on either the initiation or completion of recrystallization. However, there appears to be an over-all tendency for higher process-annealing temperatures [1925 and 2205 C (3500 and 4000 F)] to inhibit recrystallization in the final rolled condition. Figure 14, developed from the recrystallization curves (Appendix III), illustrates this behavior.

TABLE 15. TENSILE PROPERTIES OF Ta-5W-2.5Mo-0.5Zr-0.07C AT 1480 C (2700 F) SHOWING THE EFFECTS OF PRIOR FABRICATION HISTORY^(a)

| Alloy Specimen | Condition ^(b) | 1480 C (2700 F) | | Structure ^(c) | Ultimate Tensile Strength, 1000 psi | Yield Strength, 0.2 Per Cent Offset, 1000 psi | Elongation in 1/2 Inch, per cent |
|-----------------------|--|-----------------|--|--------------------------|-------------------------------------|---|----------------------------------|
| | | | | | | | |
| 315N | PR 65% at 1650 C/1 hr 1650 C/WR 50% at 425 C/1 hr 1205 C | | | W | 35.2 | 25.2 | 48 |
| 315B-L ^(d) | PR 65% at 1650 C/1 hr 1650 C/WR 50% at 425 C/1 hr 1205 C | | | W | 36.6 | 23.5 | 56 |
| 315N | PR 65% at 1650 C/1 hr 1650 C/WR 50% at 425 C/1 hr 1760 C | | | R | 38.3 ^(e) | 32.5 | 43 ^(e) |
| 315B-H ^(d) | PR 65% at 1650 C/1 hr 1650 C/PR 50% at 980 C/1 hr 1205 C | | | W | 34.6 | 22.0 | 68 |
| 315C-L | PR 65% at 1650 C/1 hr 1925 C/WR 50% at 425 C/1 hr 1205 C | | | W | 35.2 | 28.8 | 46 |
| 315C-H | PR 65% at 1650 C/1 hr 1925 C/PR 50% at 980 C/1 hr 1205 C | | | W | 38.2 | 25.0 | 20 |
| 315D-L | PR 65% at 1650 C/1 hr 2205 C/WR 50% at 425 C/1 hr 1205 C | | | W | 51.7 ^(f) | 34.3 | 6 |
| 315E-L | PR 40% at 1650 C/1 hr 1650 C/WR 75% at 425 C/1 hr 1205 C | | | W | 34.6 | 24.0 | 76 |
| 315E-H | PR 40% at 1650 C/1 hr 1650 C/PR 75% at 980 C/1 hr 1205 C | | | W | 32.2 | 23.5 | 76 |
| 315F-L | PR 40% at 1650 C/1 hr 1925 C/WR 75% at 425 C/1 hr 1205 C | | | W | 33.6 | 23.2 | 100 |
| 315K | 1 hr 2205 C/PR 75% at 1650 C/1 hr 1205 C | | | W | 42.8 | 26.5 | 40 |
| 315K | 1 hr 2205 C/PR 75% at 1650 C/1 hr 1650 C | | | R | 51.2 | 35.5 | 40 |

(a) Tested in vacuum using a mechanical screw-driven crosshead with a speed of 0.02 inch per minute for a 1/2-inch reduced section (corresponding to an approximate strain rate of 0.04 inch per inch per minute).

(b) CR = cold rolled

WR = warm rolled

PR = pack rolled.

(c) W = wrought

PW = partially wrought

R = recrystallized.

(d) Additional letter identification indicates "L" for 435 C (800 F) final rolling and "H" for 980 C (1800 F) final rolling.

(e) Average of two values.

(f) Vacuum failure. Specimen cooled, reheated, and tested. Visual examination of specimen showed evidence of severe contamination. Thus strength values probably high and elongation low.

TABLE 16. RECRYSTALLIZATION TEMPERATURES OF Ta-5W-2.5Mo-0.5Zr-0.07C FOR VARIOUS FABRICATION CONDITIONS

| Alloy Specimen | Condition (a) | Recrystallization Temperature ^(b) | |
|-----------------------|--|--|------|
| | | C | F |
| 315B-L ^(c) | PR 65% at 1650 C/1 hr 1650 C/WR 50% at 425 C | 1600 | 2910 |
| 315B-H ^(c) | PR 65% at 1650 C/1 hr 1650 C/PR 50% at 980 C | 1600 | 2910 |
| 315C-L | PR 65% at 1650 C/1 hr 1925 C/WR 50% at 425 C | 1700 | 3090 |
| 315C-H | PR 65% at 1650 C/1 hr 1925 C/PR 50% at 980 C | 1700 | 3090 |
| 315D-L | PR 65% at 1650 C/1 hr 2205 C/WR 50% at 425 C | 1700 | 3090 |
| 315D-H | PR 65% at 1650 C/1 hr 2205 C/PR 50% at 980 C | 1800 | 3270 |
| 315E-L | PR 40% at 1650 C/1 hr 1650 C/WR 75% at 425 C | 1700 | 3090 |
| 315E-H | PR 40% at 1650 C/1 hr 1650 C/PR 75% at 980 C | 1600 | 2910 |
| 315F-L | PR 40% at 1650 C/1 hr 1925 C/WR 75% at 425 C | 1700 | 3090 |
| 315F-H | PR 40% at 1650 C/1 hr 1925 C/PR 75% at 980 C | 1700 | 3090 |
| 315H-H | PR 40% at 1650 C/1 hr 2205 C/PR 75% at 980 C | 1700 | 3090 |
| 315K | 1 hr 2205 C/PR 75% at 1650 C | 1700 | 3090 |

(a) WR = warm rolled

PR = pack rolled.

(b) Complete recrystallization (75 per cent or greater) after 1-hour exposure in vacuum.

(c) Additional letter identification indicates "L" for 425 C (800 F) final rolling and "H" for 980 C (1800 F) final rolling.

TABLE 17. MICROSTRUCTURES AND HARDNESSES OF Ta-5W-2.5Mo-0.5Zr-0.07C FOR VARIOUS FABRICATION CONDITIONS(a)

| Alloy Specimen | Microstructure(b) and Hardness(c), VHN, After Annealing 1 Hour at Indicated Temperature | | | | | | | | | | |
|----------------|---|-------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| | Cast, RT | Wrought, RT | 1100 C (2010 F) | 1200 C (2190 F) | 1300 C (2370 F) | 1400 C (2550 F) | 1500 C (2730 F) | 1600 C (2910 F) | 1700 C (3090 F) | 1800 C (3270 F) | 1900 C (3450 F) |
| 315B-L(d) | -- 292 | W 370 | W 330 | W 319 | Rb 304 | Rp 251 | Rp 253 | <u>R</u> 279 | R 264 | R 274 | R 281 |
| 315B-H(d) | -- 292 | W 348 | W 327 | W 314 | Rb 299 | Rp 264 | Rp 253 | <u>R</u> 274 | R 264 | R 266 | R 289 |
| 315C-L | -- 297 | W 360 | W 354 | W 333 | W 312 | Rb 283 | Rp 279 | Rp 279 | <u>R</u> 272 | R 268 | R 285 |
| 315C-H | -- 297 | W 351 | W 333 | W 312 | Rb 309 | Rp 262 | Rp 251 | Rp 270 | <u>R</u> 270 | R 272 | R 294 |
| 315D-L | -- 297 | W 366 | W 354 | W 342 | W 322 | Rb 283 | Rp 294 | Rp 276 | <u>R</u> 274 | R 279 | R 287 |
| 315D-H | -- 297 | W 366 | W 333 | W 312 | Rb 302 | Rp 276 | Rp 264 | Rp 274 | Rp 279 | R 266 | R 287 |
| 315E-L | -- 291 | W 376 | W 345 | W 333 | Rp 306 | Rp 243 | Rp 251 | Rp 274 | <u>R</u> 264 | R 266 | R 283 |
| 315E-H | -- 291 | W 360 | W 333 | W 317 | Rb 281 | Rp 238 | Rp 249 | <u>R</u> 274 | R 274 | R 253 | R 285 |
| 315F-L | -- 295 | W 363 | W 360 | W 333 | Rb 304 | Rp 245 | Rp 249 | Rp 264 | <u>R</u> 274 | R 272 | R 297 |
| 315F-H | -- 295 | W 363 | W 345 | W 330 | Rb 312 | Rp 249 | Rp 253 | Rp 266 | <u>R</u> 272 | R 274 | R 287 |
| 315H-H | -- 292 | W 351 | W 339 | W 327 | W 304 | Rb 247 | Rp 254 | Rp 266 | <u>R</u> 266 | R 274 | R 297 |
| 315K | -- 283 | W 360 | W 319 | W 304 | W 294 | Rb 274 | Rp 253 | Rp 272 | <u>R</u> 260 | R 256 | R 276 |

(a) Wrought condition given in Table 16.

(b) W = wrought

R_b = recrystallization beginning

R_p = recrystallization partially complete

R = recrystallization essentially complete.

Minimum temperature for complete recrystallization (75 per cent or greater) is underlined.

(c) Hardness values are the average of five impressions using a 10-kg load.

(d) Additional letter identification indicates "L" for 425 C (800 F) final rolling and "H" for 980 C (1800 F) final rolling.

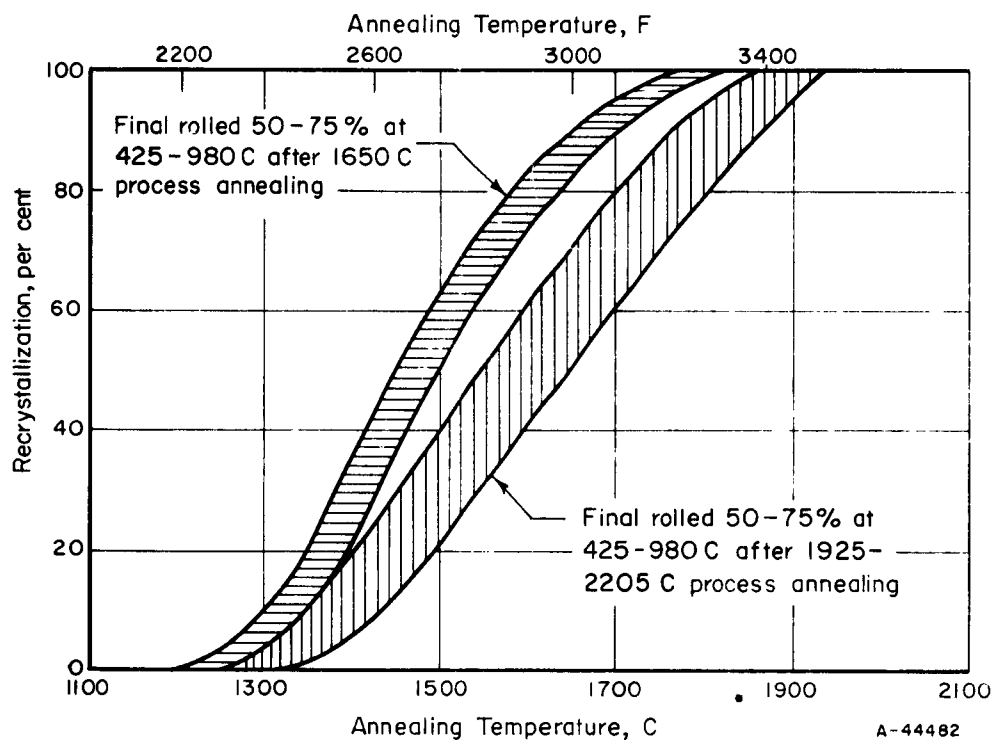


FIGURE 14. RECRYSTALLIZATION BEHAVIOR OF Ta-5W-2.5Mo-0.5Zr-0.07C, SHOWING THE EFFECT OF PROCESS-ANNEALING TEMPERATURE

A minor but very definite effect of amount of final cold reduction on the stability of grain size is shown in Table 18. Material cold worked 75 per cent produced finer grained structures than that worked only 50 per cent, and this effect was more pronounced at the higher recrystallization temperature [1900 C (3450 F)].

Thermal Exposure. Exposing the recrystallized Ta-5W-2.5Mo-0.5Zr-0.07C alloy for 10 hours at 1925 C (3500 F) in vacuum caused severe loss of both high- and low-temperature properties. Bend tests at 25 to 205 C (75 to 400 F) showed no measurable bend ductility (>16 T). High-temperature tensile tests on this exposed alloy indicated severe loss of 1480 C (2700 F) tensile properties, however at 1925 C (3500 F) strength properties were not so radically affected. Tensile ductility of the exposed material at 1925 C (3500 F) was much less than that of the unexposed alloy. These data are shown below:

| Condition | Temperature | | Tensile Strength, | Yield Strength, | Elongation, per cent |
|-------------------|-------------|------|-------------------|-----------------|----------------------|
| | C | F | 1000 psi | 1000 psi | |
| Recrystallized | 1480 | 2700 | 38.0 | 32.5 | 56 |
| Exposed | 1480 | 2700 | 27.8 | 24.5 | 1 |
| Recrystallized(a) | 1925 | 3500 | 11.6 | 10.8 | 139 |
| Exposed | 1925 | 3500 | 10.5 | 9.9 | 24 |

(a) Average data for two tests.

This loss in both high- and low-temperature properties of the Ta-5W-2.5Mo-0.5Zr-0.07C alloy after exposure may reflect altered dispersed-phase characteristics and/or contamination during exposure.

Detailed Behavior of Solid Solution Alloys

This phase of the over-all program was designed to better define the effects of the promising additions of molybdenum and tungsten on (1) high-temperature stress-rupture performance; (2) ductile-to-brittle transition behavior; and (3) welding characteristics. Alloys selected to study stress-rupture at 1480 and 1925 C (2700 and 3500 F) and bend transition behavior at -195 to 315 C (-320 to 600 F) contained tungsten and/or molybdenum contents up to 20 atomic per cent. Four alloys, Ta-5Mo, Ta-12.5W, Ta-5W-2.5Mo, and Ta-10W-2.5Mo, were selected for detailed welding investigations.

Fabrication

Alloys containing tungsten and/or molybdenum for the study of solid solution behavior were fabricated using standard fabrication practices developed and used in the past. Alloy composition and cast hardness were used as a guide to select fabrication temperatures. In general, alloys containing about 15 atomic per cent total alloying addition(s) and/or having a cast hardness above about 300 VHN were fabricated at 1650 C (3000 F). Fabrication is outlined on page 54.

TABLE 18. GRAIN SIZES OF Ta-5W-2.5Mo-0.5Zr-0.07C AFTER
HIGH-TEMPERATURE ANNEALING

| Alloy Specimen | Condition ^(a) | Average ASTM Grain Size ^(b) After Annealing 1 Hour at Indicated Temperature | |
|-----------------------|--|---|--------------------|
| | | 1700 C (3090 F) | 1900 C (3450 F) |
| 315B-L ^(c) | PR 65% at 1650 C/1 hr 1650 C/WR 50% at 425 C | 7-1/2-8 | 7 |
| 315B-H ^(c) | PR 65% at 1650 C/1 hr 1650 C/PR 50% at 980 F | 8 | 7-1/2 |
| 315C-L | PR 65% at 1650 C/1 hr 1925 C/WR 50% at 425 C | 8-1/2 | 5-1/2 |
| 315C-H | PR 65% at 1650 C/1 hr 1925 C/PR 50% at 980 C | 7-1/2 | 5-1/2 |
| 315D-L | PR 65% at 1650 C/1 hr 2205 C/WR 50% at 425 C | 7-1/2-8 | 5-1/2 |
| 315D-H | PR 65% at 1650 C/1 hr 2205 C/PR 50% at 980 C | 8 | 6-1/2 |
| 315E-L | PR 40% at 1650 C/1 hr 1650 C/WR 75% at 425 C | 8-1/2 | 7-1/2 |
| 315E-H | PR 40% at 1650 C/1 hr 1650 C/PR 75% at 980 C | 8 | 7-1/2 |
| 315F-L | PR 40% at 1650 C/1 hr 1925 C/WR 75% at 425 C | 8-1/2 | 8 |
| 315F-H | PR 40% at 1650 C/1 hr 1925 C/PR 75% at 980 C | 9 | 7-1/2-8 |
| 315H-H | PR 40% at 1650 C/1 hr 2205 C/PR 75% at 980 C | 8 | 7 |
| 315K | 1 hr 2205 C/PR 75% at 1650 C | 7-1/2 | 7 |

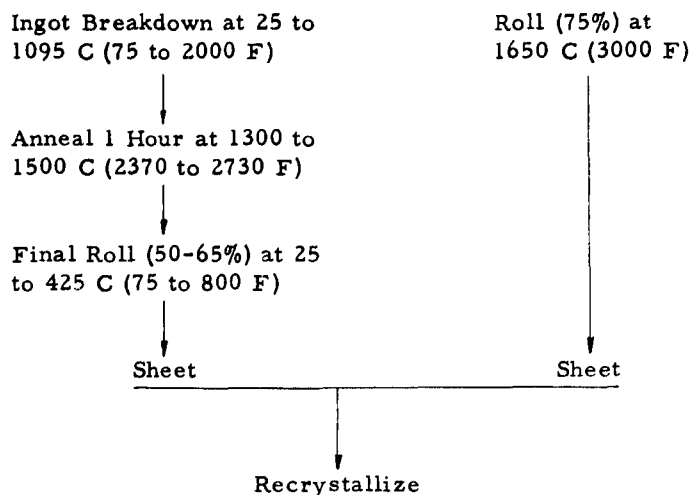
(a) CR = cold rolled

WR = warm rolled

PR = pack rolled.

(b) Obtained by comparison with ASTM grain size chart at 100X.

(c) Additional letter identification indicates "L" for 425 C (800 F) final rolling and "H" for 980 C (1800 F) final rolling.



Fabrication data for solid solution strengthened alloys, Table 19, indicate excellent correlation and agreement with past results⁽³⁾. All alloys fabricated at low temperatures [25 to 425 C (75 to 800 F)] were rolled to excellent-quality sheet except Specimen 268F which failed (inexplicably) after about 10 per cent reduction. Specimen 268H replaced Specimen 268F.

A number of alloys were fabricated using an ingot breakdown temperature of 1095 C (2000 F), a 1-hour 1500 C (2730 F) intermediate recrystallization treatment, and final reductions of 50 to 55 per cent at 260 to 425 C (500 to 800 F). These include Ta-5Mo (six ingots), Ta-10W (two ingots), Ta-12.5W (three ingots), and Ta-5W-5Mo (two ingots) compositions. All materials were fabricated to excellent quality sheet except Ta-5W-5Mo. The Ta-5W-5Mo alloy exhibited some tendency toward 45-degree surface cracking (relative to the rolling direction) during sheet rolling at 425 C (800 F), indicating that final rolling temperature should be higher for this alloy because of the relatively large content of tungsten and molybdenum - about 14 atomic per cent. However, cracking was not severe enough to preclude test-sample preparation.

Most alloys that were rolled directly to sheet from cast ingots at 1650 C (3000 F) were of excellent quality. In two alloy strips, Ta-10W-5Mo and Ta-15W-2.5Mo, the as-rolled surface exhibited highly distorted and elongated grains and showed minor cracking at grain boundaries. Alloys of this type (containing about 18 to 19 atomic per cent tungsten and/or molybdenum) closely approach the previously established alloy limits for fabricability for Ta-W, Ta-Mo, and Ta-W-Mo alloys⁽³⁾.

Evaluation

The evaluation of Ta-Mo, Ta-W, and Ta-W-Mo alloys for high-temperature (stress-rupture and thermal exposure), low-temperature (bend transition), and welding behavior was conducted for all materials in the recrystallized condition.

TABLE 19. FABRICATION DATA FOR SOLID SOLUTION STRENGTHENED TANTALUM-BASE ALLOYS

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Cast Hardness, (a) UHN | Initial Rolling Tempera- ture (b) | | Quality of Strip (c) | Intermediate Annealing Temperature | | Final Rolling Tempera- ture (b) | | Final Reduction, per cent | Quality of Strip (c) |
|---|-------------------|------------------------------|---|------|-------------------------|--|------|---------------------------------------|-----|---------------------------------|-------------------------|
| | | | C | F | | C | F | C | F | | |
| | | | Transition Behavior | | | | | | | | |
| 2.5Mo | 322 | 157 | 370 | 700 | Excellent | 1400 | 2550 | 25 | 75 | 65 | Excellent |
| 5Mo | 99E | 217 | 1095 | 2000 | Excellent | 1500 | 2730 | 425 | 800 | 55 | Excellent |
| 7.5Mo | 161D | 262 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 10Mo | 100E | 308 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 10W | 88D | 219 | 1095 | 2000 | Excellent | 1500 | 2730 | 260 | 500 | 55 | Excellent |
| 15W | 165C | 261 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 20W | 89G | 304 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 5.2W-2.7Mo | 323 | 225 | 425 | 800 | Excellent | 1400 | 2550 | 25 | 75 | 60 | Excellent |
| 7.9W-4.1Mo | 324 | 280 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 10.6W-5.6Mo | 325 | 315 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| Stress-Rupture Evaluation | | | | | | | | | | | |
| 100Ta | 191 | 88 | 25 | 75 | Excellent | 1300 | 2370 | 25 | 75 | 65 | Excellent |
| 2.5Mo | 322A | 149 | 370 | 700 | Excellent | 1400 | 2550 | 25 | 75 | 60 | Excellent |
| 2.5Mo | 322B | 148 | 370 | 700 | Excellent | 1400 | 2550 | 25 | 75 | 60 | Excellent |
| 5Mo | 99F | 218 | 1095 | 2000 | Excellent | 1500 | 2730 | 425 | 800 | 55 | Excellent |
| 5Mo | 99G | 215 | 1095 | 2000 | Excellent | 1500 | 2730 | 425 | 800 | 55 | Excellent |
| 7.5Mo | 161E | 253 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 10Mo | 100F | 302 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 5W | 164C | 155 | 25 | 75 | Excellent | 1400 | 2550 | 25 | 75 | 60 | Excellent |
| 10W | 88E | 210 | 1095 | 2000 | Excellent | 1500 | 2730 | 260 | 500 | 55 | Excellent |
| 20W | 89E | 312 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 20W | 89F | 299 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 5W-2.5Mo | 268C | 216 | 370 | 700 | Excellent | 1400 | 2550 | 25 | 75 | 60 | Excellent |
| 5W-5Mo | 277C | 260 | 1095 | 2000 | Excellent | 1500 | 2730 | 425 | 800 | 55 | Good(d) |
| 5W-5Mo | 277D | 268 | 1095 | 2000 | Excellent | 1500 | 2730 | 425 | 800 | 55 | Good(d) |
| 5W-7.5Mo | 331 | 315 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 5W-7.5Mo | 331A | 313 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 10W-2.5Mo | 262C | 278 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 10W-2.5Mo | 262D | 273 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 10W-5Mo | 179F | 308 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Good(e) |
| 10W-5Mo | 179G | 306 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 15W-2.5Mo | 326 | 320 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 15W-2.5Mo | 326A | 350 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Fair(f) |
| Welding Variables | | | | | | | | | | | |
| 5Mo | 99H | 215 | 1095 | 2000 | Excellent | 1500 | 2730 | 425 | 800 | 55 | Excellent |
| 5Mo | 99I | 215 | 1095 | 2000 | Excellent | 1500 | 2730 | 425 | 800 | 55 | Excellent |
| 5Mo | 99J | 218 | 1095 | 2000 | Excellent | 1500 | 2730 | 425 | 800 | 55 | Excellent |
| 12.5W | 266B | 242 | 1095 | 2000 | Excellent | 1500 | 2730 | 370 | 700 | 50 | Excellent |
| 12.5W | 266C | 242 | 1095 | 2000 | Excellent | 1500 | 2730 | 370 | 700 | 50 | Excellent |
| 12.5W | 266D | 240 | 1095 | 2000 | Excellent | 1500 | 2730 | 370 | 700 | 50 | Excellent |
| 5W-2.5Mo | 268D | 221 | 370 | 700 | Excellent | 1400 | 2550 | 25 | 75 | 55 | Excellent |
| 5W-2.5Mo | 268E | 222 | 370 | 700 | Excellent | 1400 | 2550 | 25 | 75 | 55 | Excellent |
| 5W-2.5Mo | 268F | 224 | 370 | 700 | Poor | -- | -- | -- | -- | 10 | -- |
| 5W-2.5Mo | 268H | 210 | 370 | 700 | Excellent | 1400 | 2550 | 25 | 75 | 55 | Excellent |
| 10W-2.5Mo | 262E | 266 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 10W-2.5Mo | 262F | 270 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |
| 10W-2.5Mo | 262G | 258 | 1650 | 3000 | -- | -- | -- | -- | -- | 75 | Excellent |

(a) Hardness values are the average of five impressions using a 10-kg load.

(b) Alloys rolled at 260 to 425 C (500 to 800 F) unprotected in air. Alloys rolled at 1095 C (2000 F) in evacuated stainless steel packs. Alloys rolled at 1650 C (3000 F) in evacuated molybdenum packs.

(c) Excellent - no cracking of edges or surface

Good - slight cracking of edges and surface

Fair - considerable cracking of edges and surface.

(d) Some 45-degree surface cracking noted.

(e) Few surface cracks along highly distorted grain boundaries. Broke on machining stress-rupture specimens.

(f) Numerous surface cracks along highly distorted grain boundaries.

Transition Behavior. The effects of temperature on the bend ductility of selected recrystallized Ta-Mo, Ta-W, and Ta-W-Mo alloys are given in Table 20 and illustrated in Figure 15. By selecting the temperature required to give a 4T minimum bend radius (from Figure 15) and replotting versus alloy content, the effects of Group VI-A metal additions to tantalum were established as shown in Figure 16. These data clearly show a more drastic effect of molybdenum additions compared to the effect of tungsten additions on the low-temperature ductility of tantalum. For example, at 14 atomic per cent binary addition, the tungsten-containing alloy exhibits a 4T transition temperature of about -200 C (-330 F), whereas the value for the molybdenum-containing alloy is about -100 C (-150 F). As the alloy concentration increases, the inferiority of molybdenum becomes more pronounced. Figure 16 further suggests that in high-purity alloys, tantalum exhibits a large tolerance for molybdenum or tungsten additions (12 to 13 atomic per cent) concurrent with excellent cryogenic ductility at temperatures as low as -250 C (-420 F). As expected, Ta-W-Mo alloys containing at least 15 atomic per cent combined addition (1:1 tungsten-molybdenum atomic concentration ratio) fall between the limits established for binary additions.

The transition behavior characteristics established for these tantalum-base alloys strengthened with molybdenum and/or tungsten can be considered representative of laboratory-produced button ingots having a low total interstitial content (50 to 100 ppm max). As interstitial content increases, less tolerance for molybdenum and/or tungsten would be expected. However, the data show that up to 19 per cent tungsten can be added to tantalum and still maintain 4T bend ductility at room temperature. These data demonstrate greater tolerances for molybdenum and tungsten than previously reported⁽³⁾ based on screening tests.

Stress-Rupture Behavior. Stress-rupture data for recrystallized tantalum, Ta-Mo, Ta-W, and Ta-W-Mo, alloys at 1480 C (2700 F) are presented, along with selected data from previous work⁽³⁾, in Table 21.

Stress-rupture curves for binary Ta-Mo and Ta-W alloys, Figures 17 and 18, respectively, show progressive increase in rupture parameters with increasing molybdenum or tungsten contents. Similar behavior is noted for ternary Ta-W-Mo alloys, as shown in Figure 19.

From these curves the estimated 1- and 10-hour rupture strengths have been established as given in Table 22. Strengthening improvements, relative to unalloyed tantalum, range from about two- to sixfold.

Ta-20W exhibited the highest rupture strength at 1480 C (2700 F). The 1- and 10-hour rupture strengths, 28,500 and 19,800 psi, respectively, are superior to values for the strongest tantalum-base alloy (Ta-10-5V) tested in the past⁽³⁾, and Ta-20W is at least 30 per cent stronger than Ta-10W.

Table 23 presents stress-rupture data for recrystallized tantalum, Ta-Mo, Ta-W, and Ta-W-Mo alloys at 1925 C (3500 F).

Rupture parameters at 1925 C (3500 F) increase with increasing alloy content for molybdenum, tungsten, and combinations of these Group VI-A metal additions as illustrated in Figures 20 through 22.

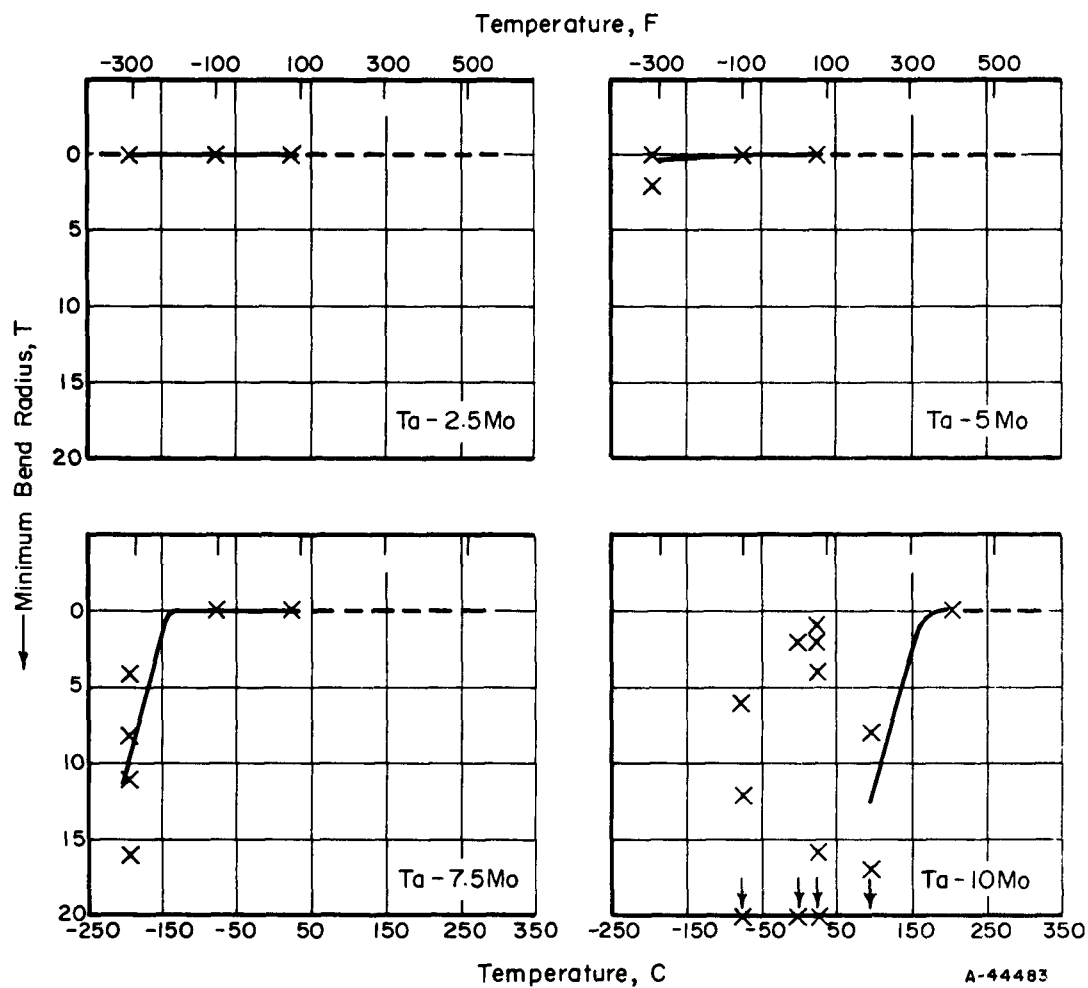


FIGURE 15. EFFECT OF TEMPERATURE ON THE BEND DUCTILITY OF RECRYSTALLIZED Ta-Mo, Ta-W, AND Ta-W-Mo ALLOYS

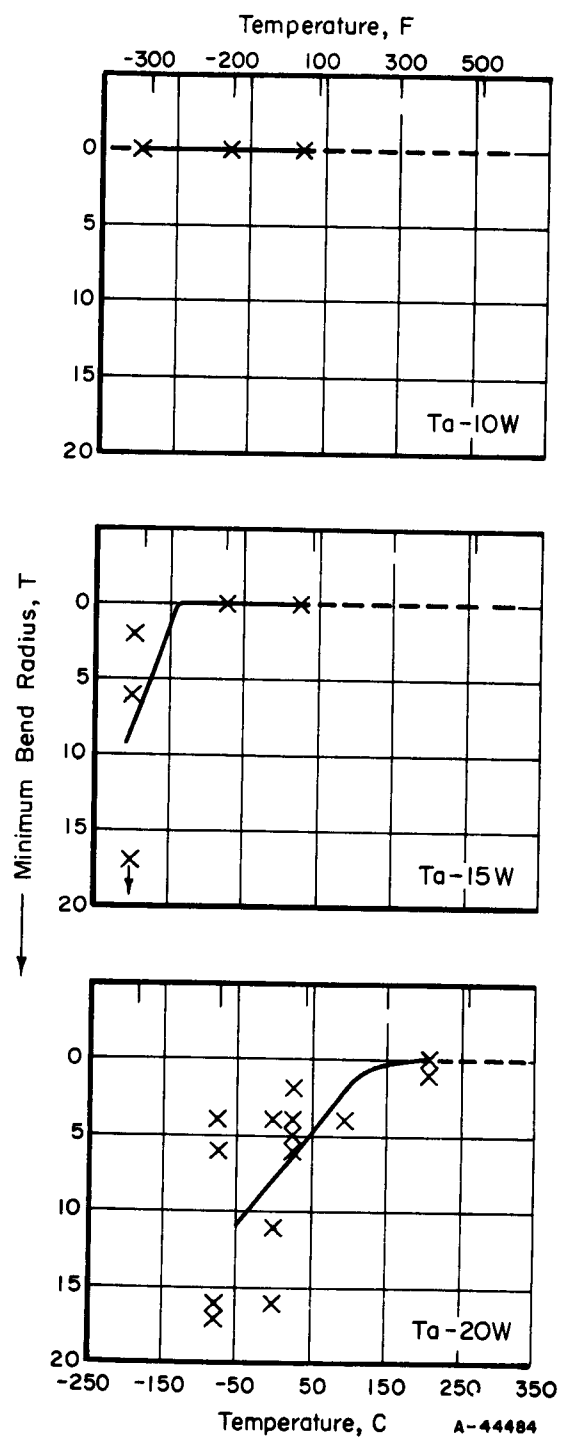


FIGURE 15. (CONTINUED)

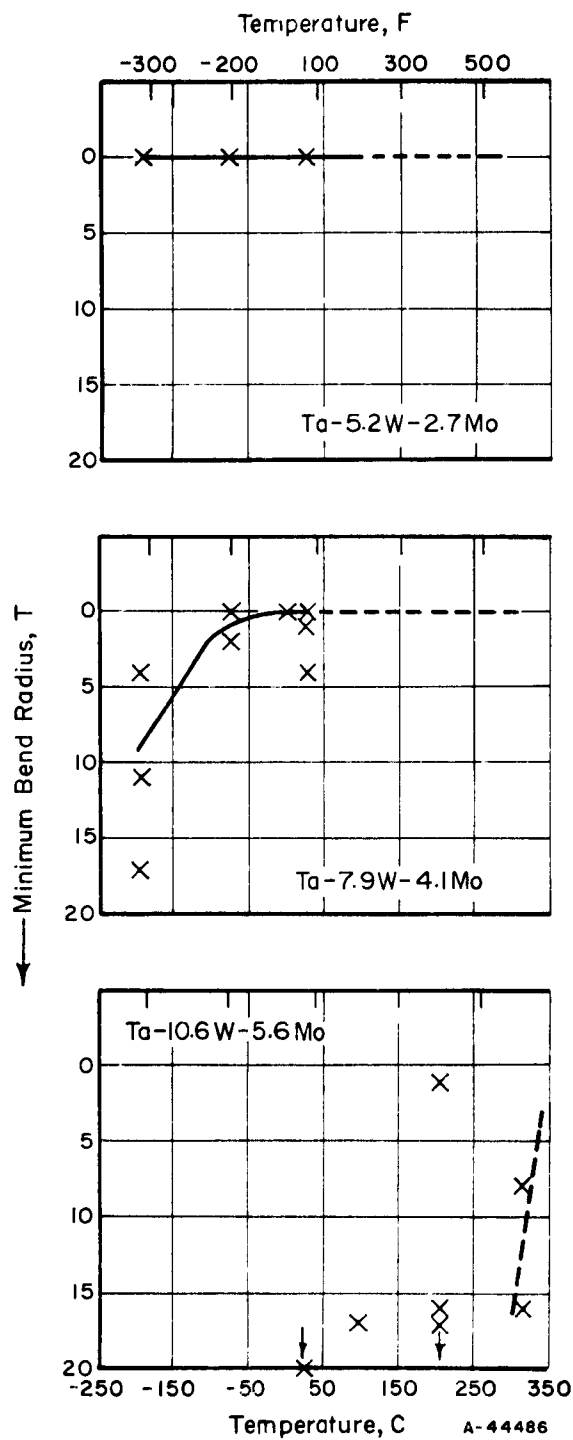


FIGURE 15. (CONTINUED)

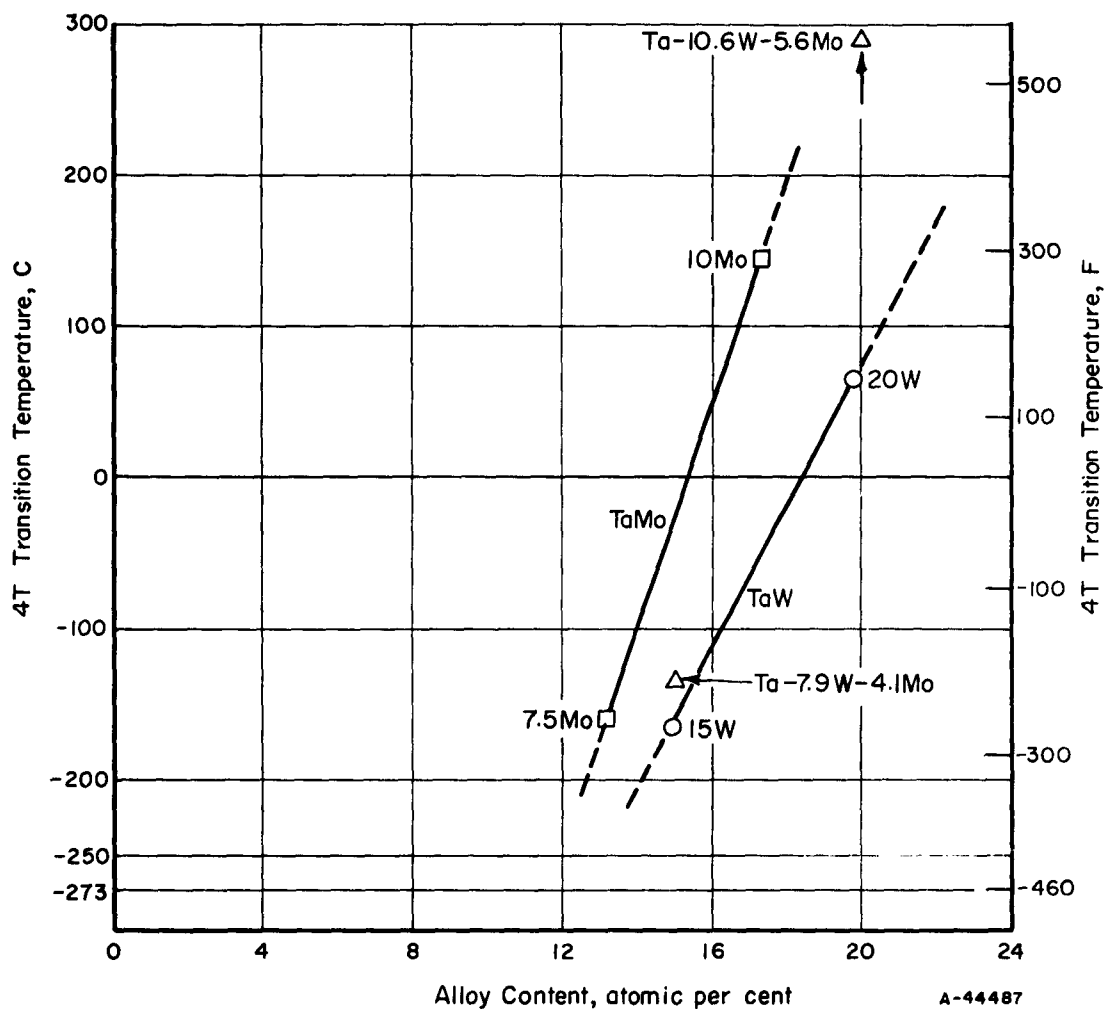


FIGURE 16. 4T TRANSITION TEMPERATURE VERSUS ALLOY CONTENT FOR RECRYSTALLIZED TANTALUM-BASE ALLOYS CONTAINING MOLYBDENUM AND TUNGSTEN

TABLE 20. EFFECT OF TEMPERATURE ON THE BEND DUCTILITY OF Ta-Mo, Ta-W, AND Ta-W-Mo ALLOYS^(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Minimum Bend Radius Value, $r^{(b)}$, at Indicated Temperature | | | | | |
|---|--------------------|---|----------------|-----------------|--------------------|--------------|------------------|
| | | -195 C (-320 F) | -75 C (-105 F) | 0 C (32 F) | 25 C (75 F) | 95 C (200 F) | 205 C (400 F) |
| 2.5Mo | 322 | 0, 0, 0, 0 | 0, 0, 0, 0 | -- | 0, 0, 0, 0 | -- | -- |
| 5Mo | 99E | 0, 0, 0, 2 | 0, 0, 0, 0 | -- | 0, 0, 0, 0 | -- | -- |
| 7.5Mo | 161D | 4, 8, 11, 16 | 0, 0, 0, 0 | -- | 0, 0, 0, 0 | -- | -- |
| 10Mo | 100E | -- | 6, 12, >35, 37 | 2, 2, >37, >42 | 1, 2, 4, >16, 44 | 8, >17 | 0, 0, 0, 0 |
| 10W | 88D | 0, 0, 0, 0 | 0, 0, 0, 0 | -- | 0, 0, 0, 0 | -- | -- |
| 15W | 165C | 2, 2, 6, >17 | 0, 0, 0, 0 | -- | 0, 0, 0, 0 | -- | -- |
| 20W | 89G | -- | 4, 6, 16, 17 | 4, 4, 4, 11, 16 | 2, 4, 5, 6 | 4, 4, 4, 4 | 0, 0, 0, 1 |
| 5.2W-2.7Mo | 323 | 0, 0, 0, 0 | 0, 0, 0, 0 | -- | 0, 0, 0, 0 | -- | -- |
| 7.9W-4.1Mo | 324 | 4, 4, 11, 17 | 0, 0, 2, 2 | 0, 0, 0, 0 | 0, 1, 1, 4 | -- | -- |
| 10.6W-5.6Mo | 325 ^(c) | -- | -- | -- | >33, >33, >33, >33 | >17, >17 | 1, >16, >17, >17 |

(a) Recrystallized material.

(b) r -value is radius of last good die before evidence of cracking appears, divided by specimen thickness.

(c) r -values of 8, 16 at 315 C (600 F).

TABLE 21. STRESS-RUPTURE PROPERTIES OF TANTALUM AND
TANTALUM-BASE ALLOYS AT 1480 C (2700 F)^(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Stress, 1000 psi | Deformation Properties | | Rupture Properties | | |
|---|-------------------|---------------------|---------------------------------------|---|---------------------------|---------------------------|---|
| | | | Elongation on Loading, per cent | Minimum Creep Rate, per cent/hour | Rupture Time, hours | Gage Length, inches | Elongation in Gage Length, per cent |
| 100Ta | (b) | 5.26 | -- | -- | 0.4 ^(c) | 1.20 | 54 |
| | (b) | 4.75 | 2.02 | -- | 0.37 | 1.20 | 42 |
| | (b) | 4.2 | 0.80 | 4.8 | 3.3 ^(d) | 1.20 | 68 |
| | (b) | 3.9 | 0.84 | 2.5 | 7.6 | 1.20 | 74 |
| 2.5Mo | 322A-1 | 11.05 | 1.09 | 31.5 | 0.96 | 1.00 | 61 |
| | 322A-2 | 8.0 | 0.43 | 5.36 | 5.55 | 1.00 | 68 |
| | 322A-3 | 7.1 | 0.04 | 2.38 | 10.92 | 1.00 | 73 |
| 5Mo | 99F-1 | 20.0 | 8.5 | -- | 0.05 | 1.00 | 30 |
| | 99F-2 | 12.0 | 1.65 | 6.97 | 3.48 | 1.00 | 49 |
| 10Mo | 100F-1 | 25.0 | 1.28 | 10.7 | 0.48 | 1.25 | 9 |
| | 100F-2 | 20.0 | 1.0 | 4.2 | 1.6 | 1.25 | 9 |
| 5W | (b) | 13.0 | 2.12 | -- | 0.35 | 1.20 | 28 |
| | (b) | 10.0 | 1.09 | 7.4 | 4.1 | 1.25 | 47 |
| | (b) | 9.0 | 0.45 | 1.6 | 17.4 | 1.25 | 47 |
| 10W | (b) | 19.0 | 1.29 | 5.3 | 1.93 | 1.24 | 19 |
| | (b) | 16.5 | 1.08 | 3.5 | 4.83 | 1.24 | 28 |
| 20W | 89E-1 | 27.0 | 1.56 | 6.76 | 1.35 | 1.25 | 12 |
| 5W-2.5Mo | (b) | 25.0 | 9.48 | -- | 0.023 | 1.25 | 33 |
| | (b) | 15.0 | 1.05 | 5.44 | 2.7 | 1.25 | 37 |
| | 268C-1 | 13.0 | 0.95 | 4.75 | 4.1 | 1.00 | 35 |
| 5W-5Mo | 227C-1 | 20.0 | 1.58 | 3.32 | 0.55 | 1.01 | 47 |
| | 227C-2 | 13.0 | 1.15 | -- | 0.8 ^(e) | 1.04 | 5 |
| 5W-7.5Mo | 331-1 | 15.0 | 0.06 | -- | 0.023 ^(e) | 1.25 | 3 |
| | 331-2 | 20.0 | 0.17 | -- | 0.108 ^(e) | 1.25 | 2 |
| 7.5W-2.5Mo | (b) | 17.0 | 1.13 | 5.7 | 2.93 | 1.24 | 57 |
| | (b) | 15.0 | 0.84 | 1.67 | 12.82 | 1.23 | 29 |
| 10W-2.5Mo | 262C-1 | 25.0 | 1.40 | 28.5 | 0.4 | 1.25 | 18 |
| | 262C-2 | 15.0 | 0.88 | 0.50 | 11.65 | 1.25 | 10 |
| 15W-2.5Mo | 326A-1 | (f) | -- | -- | -- | 1.25 | -- |

(a) Recrystallized material.

(b) Data from Reference 3.

(c) Tested at 1440 C (2620 F).

(d) Tantalum heater element failure after 23 minutes; element replaced and specimen reloaded. Rupture time is the total for both tests.

(e) Failed through flaw.

(f) Failed through grip pinhole.

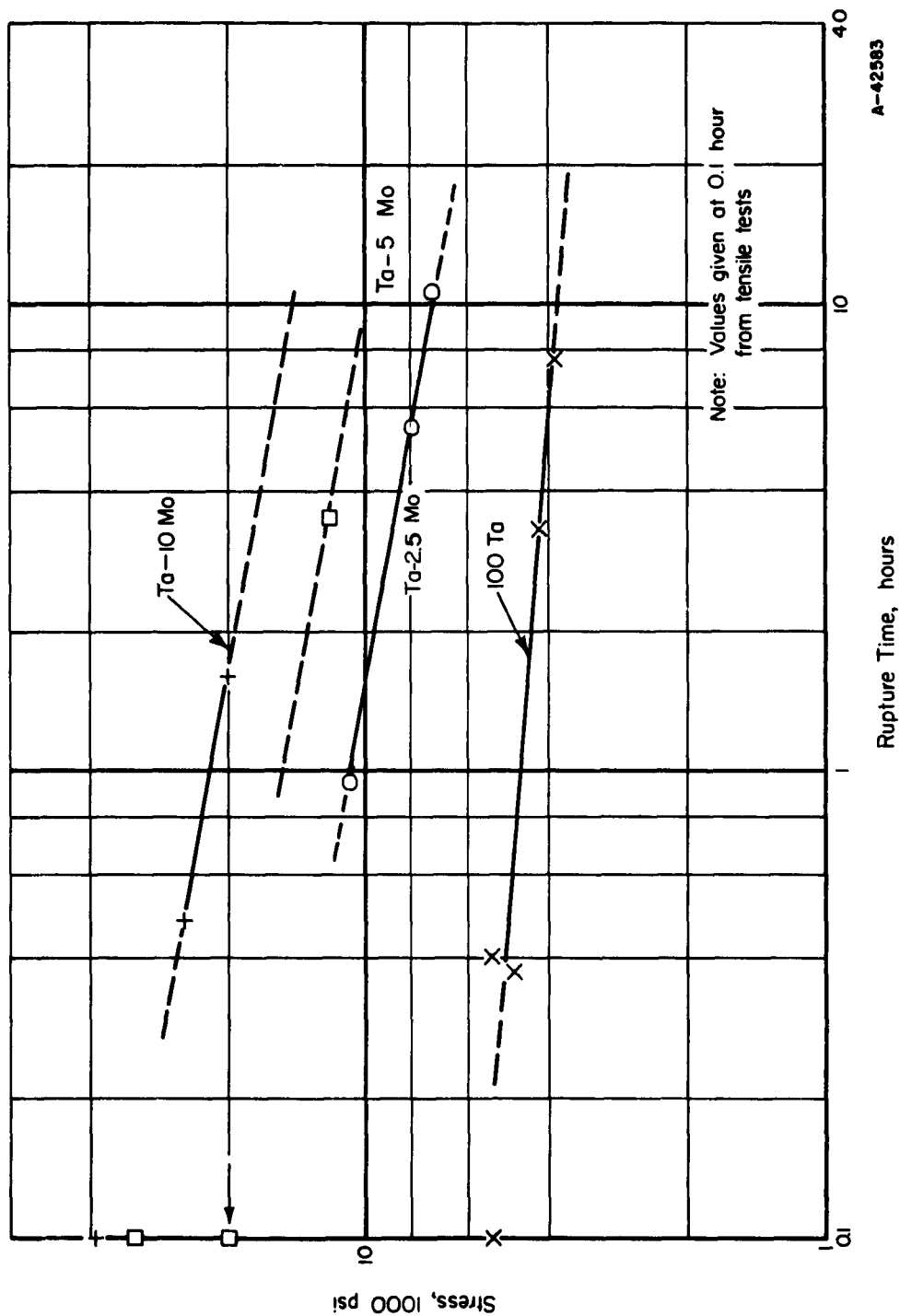


FIGURE 17. STRESS-RUPTURE CURVES FOR RECRYSTALLIZED TANTALUM AND
Ta-(2.5, 5, 10)Mo AT 1480 C (2700 F)

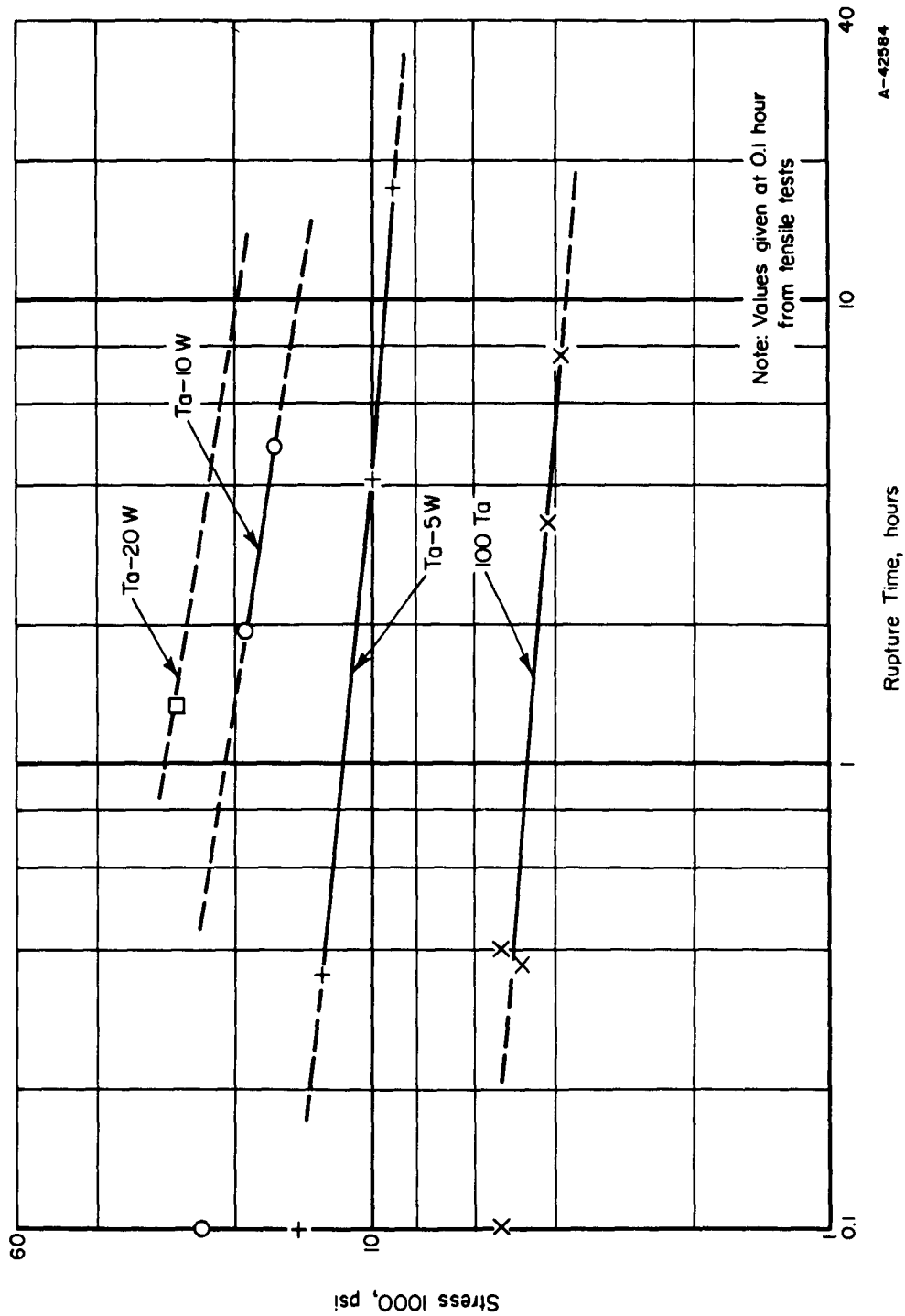


FIGURE 18. STRESS-RUPTURE CURVES FOR RECRYSTALLIZED TANTALUM AND Ta-(5, 10, 20)W AT 1480 C (2700 F)

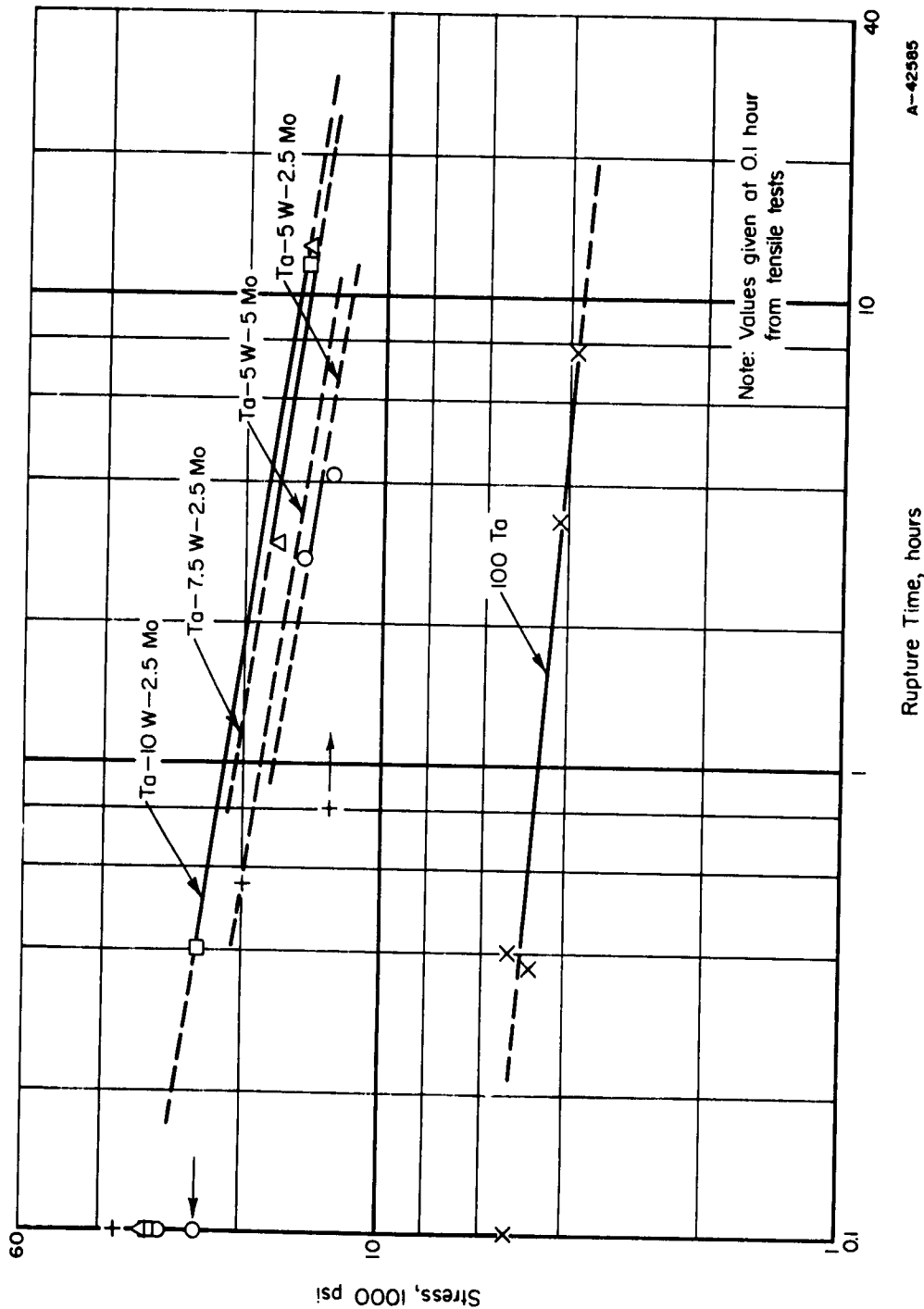


FIGURE 19. STRESS-RUPTURE CURVES FOR RECRYSTALLIZED TANTALUM, Ta-5W-(2.5, 5)Mo, AND Ta-(7.5, 10)W-2.5Mo AT 1480 C (2700 F)

TABLE 22. RUPTURE STRENGTHS OF TANTALUM AND TANTALUM-BASE ALLOYS AT 1480 C (2700 F)^(a)

| Alloy Composition (Balance Tantalum), weight per cent | Ultimate Tensile Strength, 1000 psi | Stress to Rupture ^(b) , 1000 psi, at Indicated Time | |
|---|--|--|---------|
| | | 1 Hour | 10 Hour |
| 100TA ^(c) | 5.3 | 4.6 | 3.8 |
| 2.5Mo | -- | 11.0 | 7.2 |
| 5Mo | 31.6 ^(c) | 15.2 | 10.0 |
| 10Mo | 39.4 ^(c) | 22.0 | 14.5 |
| 5W ^(c) | 14.8 | 11.7 | 9.4 |
| 10W ^(c) | 23.8 | 21.0 | 14.7 |
| 20W | -- | 28.5 | 19.8 |
| 5W-2.5Mo | 30.3 ^(c) | 17.0 | 12.1 |
| 5W-5Mo | 37.1 ^(c) | 18.2 | 13.0 |
| 7.5W-2.5Mo | 32.2 ^(c) | 20.8 | 14.8 |
| 10W-2.5Mo | 31.4 ^(c) | 21.9 | 15.5 |

(a) Recrystallized material.

(b) Rupture strengths obtained graphically from a stress-rupture plot.

(c) Data from Reference 3.

TABLE 23. STRESS-RUPTURE PROPERTIES OF TANTALUM AND TANTALUM-BASE ALLOYS AT 1925 C (3500 F)^(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Stress, 1000 psi | Deformation Properties | | Rupture Properties | | |
|---|-------------------|------------------------|---------------------------------------|---|---------------------------|---------------------------|---|
| | | | Elongation on Loading, per cent | Minimum Creep Rate, per cent/hour | Rupture Time, hours | Gage Length, inches | Elongation in Gage Length, per cent |
| 100Ta | 191-5 | 1.5 | 0.48 | 0.679 | 7.5 ^(b) | 1.00 | 49 |
| | 191-6 | 2.2 | -- | -- | 0.034 | 1.00 | 53 |
| | 191-7 | 1.8 | 3.65 | -- | 0.05 | 1.00 | 62 |
| | 191-8 | 1.6 | 2.42 | -- | 0.092 | 1.00 | 43 |
| 2.5Mo | 322B-1 | 2.8 | 0.015 | 6.2 | 4.6 | 1.00 | 56 |
| | 322B-2 | 2.833 | 0.095 | 4.64 | 7.0 | 1.00 | 91 |
| | 322B-3 | 3.6 | 0.19 | 18.0 | 1.6 | 1.00 | 90 |
| | 322B-4 | 4.2 | 0.25 | 57.1 | 0.55 | 1.00 | 100 |
| 5Mo | 99G-1 | 5.0 | 0.048 | 6.8 | 2.3 | 1.03 | 111 |
| | 99G-2 | 4.0 | 0.071 | 5.2 | 5.9 | 1.05 | 86 |
| 7.5Mo | 161E-1 | 8.0 | 0.120 | 7.9 | 0.35 | 1.25 | 70 |
| | 161E-2 | 4.5 | 0.084 | 6.25 | 6.06 | 1.25 | 42 |
| 5W | 164C-1 | 6.0 | 3.29 | 292 | 0.134 | 1.00 | 81 |
| | 164C-2 | 3.0 | 0.002 | 4.7 | 9.27 | 1.00 | 74 |
| | 164C-3 | 5.0 | 0.13 | 80 | 0.45 | 1.00 | 80 |
| | 164C-4 | 4.5 | 0.89 | 50 | 0.78 | 1.00 | 81 |
| 10W | 88E-1 | 8.0 | 0.044 | 36.1 | 0.53 | 1.03 | 57 |
| | 88E-2 | 5.6 | 0.0096 | 7.3 | 3.4 | 1.04 | 63 |
| 20W | 89F-1 | 10.0 | 0.044 | 16.97 | 0.84 | 1.25 | 19 |
| | 89F-2 | 6.0 | 0.04 | 5.05 | 5.95 ^(c) | 1.25 | 13 |
| 5W-2.5Mo | 268C-2 | 6.2 | 0.23 | 162.5 | 0.5 | 1.00 | 86 |
| | 268C-3 | 3.5 | 0.015 | 2.55 | 12.2 | 1.00 | 98 |
| | 268C-4 | 4.5 | 0 | 15.59 | 2.63 | 1.00 | 79 |
| 5W-5Mo | 227D-1 | 7.7 | 0.18 | 6.32 | 0.43 | 1.05 | 63 |
| | 227D-2 | 4.5 | 0.234 | 3.59 | 5.25 | 1.00 | 36 |
| 5W-7.5Mo | 331A-1 | 8.0 | 0.168 | -- | 0.1 ^(c) | 1.25 | 0 |
| | 331A-2 | 4.6 | 0.156 | 3.66 | 2.3 ^(d) | 1.25 | 10 |
| 10W-2.5Mo | 262D-1 | 9.0 | 0 | 92.5 | 0.42 | 1.25 | 22 |
| | 262D-2 | 4.5 | 0 | 2.68 | 4.65 ^(d) | 1.25 | 13 |
| 10W-5Mo | 179G-1 | 9.0 | 0 | 13.5 | 0.24 ^(e) | 1.25 | 8 |
| | 179G-2 | 4.0 | 0 | 0.60 | 1.03 ^(e) | 1.25 | 4 |
| 15W-2.5Mo | 326-1 | (f) | -- | -- | -- | 1.25 | -- |
| | 326-2 | (f) | -- | -- | -- | 1.25 | -- |

(a) Recrystallized material.

(b) Hardness values after test indicate specimen contaminated during test.

(c) Failed through grip pinhole.

(d) Tear fracture.

(e) Many tears and cracks in specimen.

(f) Failed during assembly in stress-rupture unit.

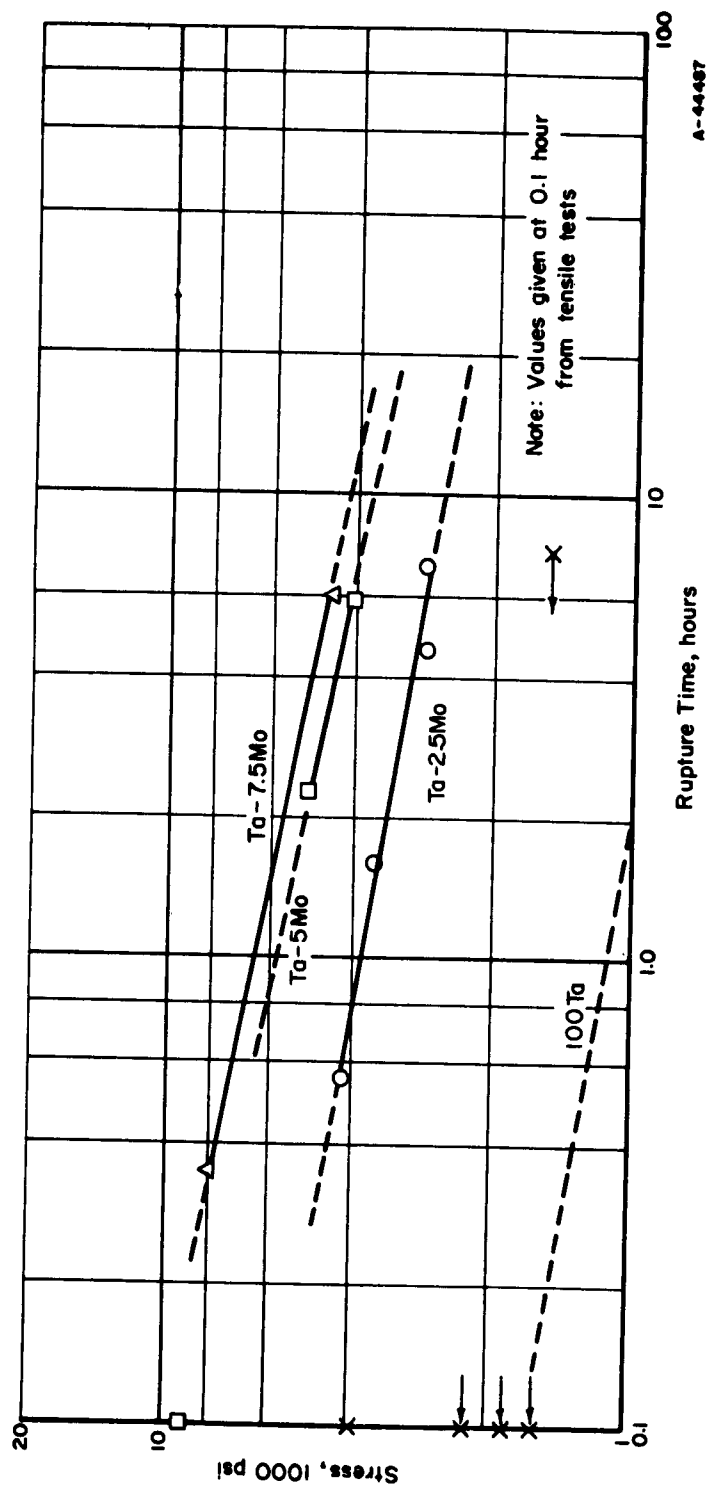


FIGURE 20. STRESS-RUPTURE CURVES FOR RECRYSTALLIZED TANTALUM AND Ta-(2.5, 5, 7.5)Mo AT 1925 C (3500 F)

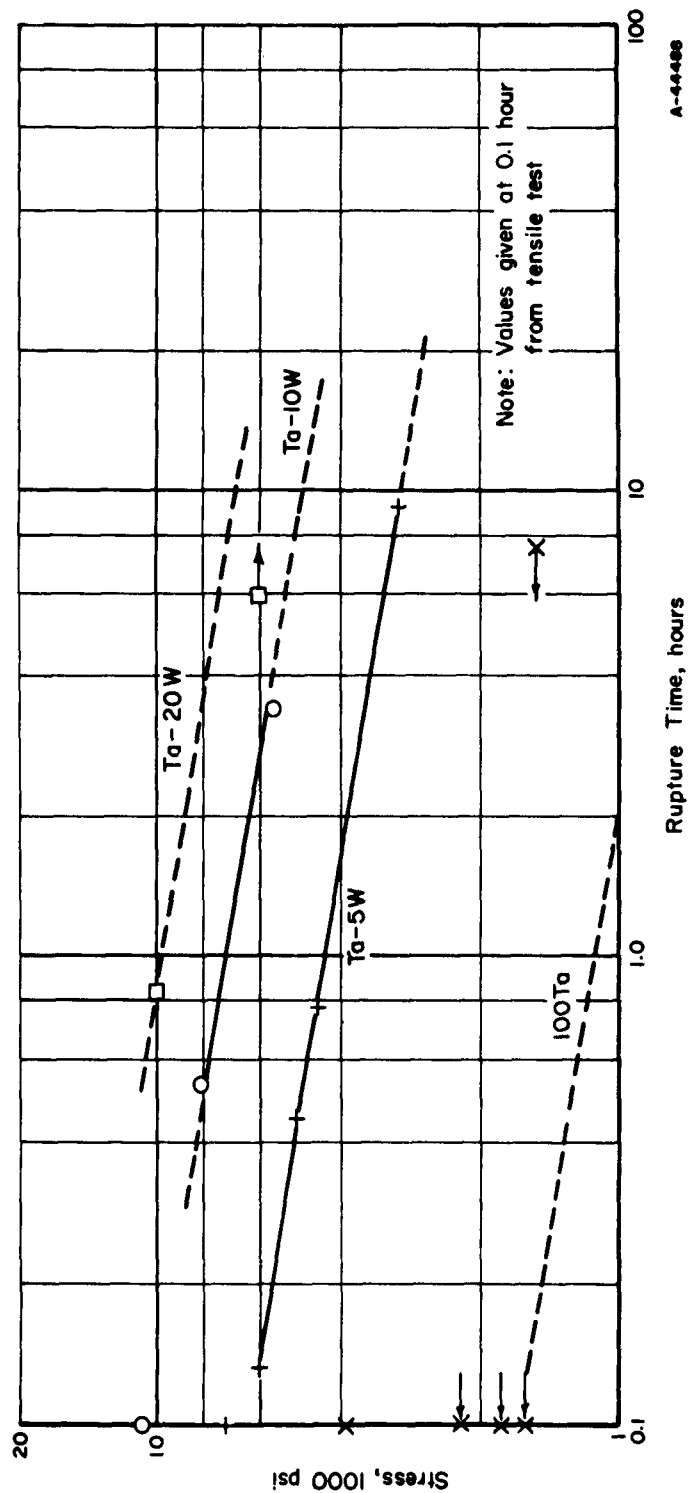


FIGURE 21. STRESS-RUPTURE CURVES FOR RECRYSTALLIZED TANTALUM AND Ta-(5, 10, 20)W AT 1925 C (3500 F)

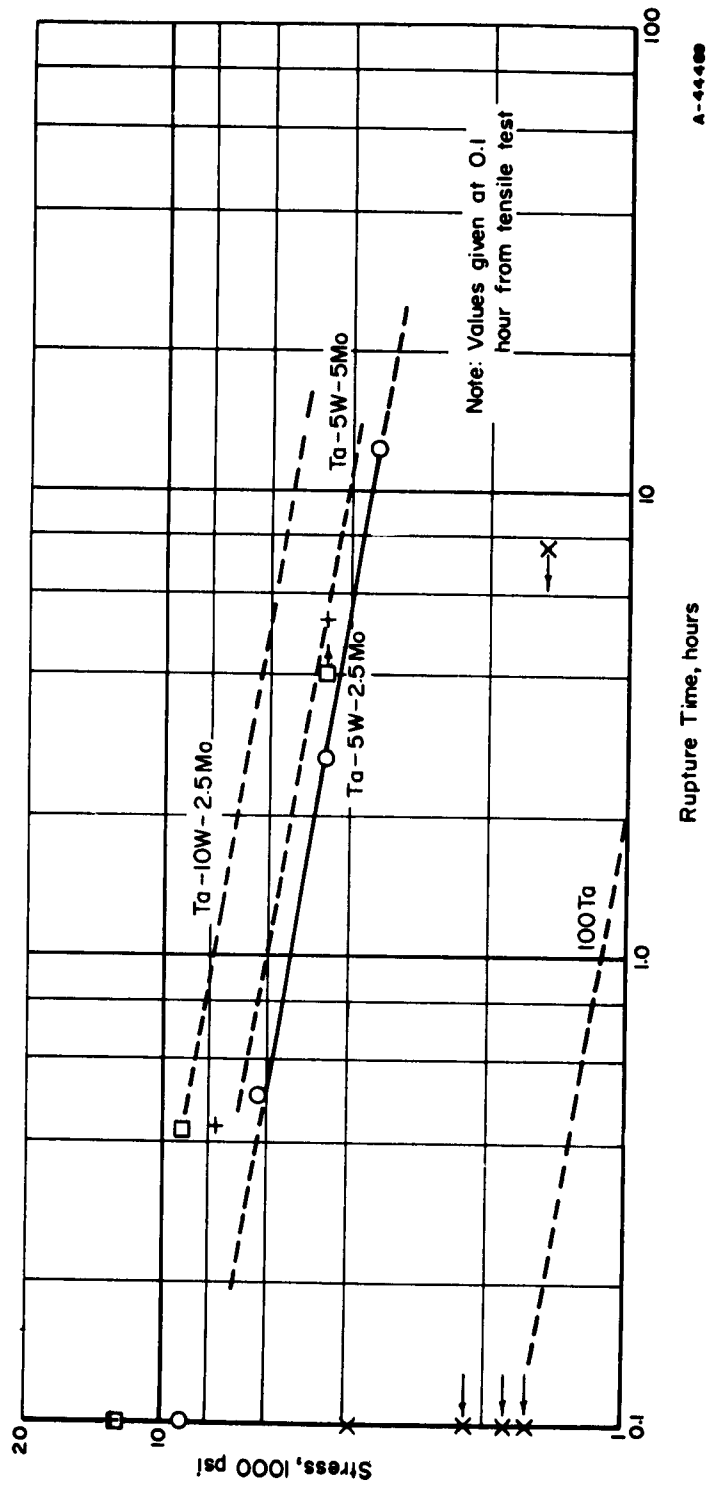


FIGURE 22. STRESS-RUPTURE CURVES FOR RECRYSTALLIZED TANTALUM, Ta-5W-(2.5, 5)Mo, AND Ta-10W-2.5Mo AT 1925 C (3500 F)

The 1- and 10-hour rupture strengths at 1925 C (3500 F) have been estimated and are given in Table 24. Improvements in rupture strength, compared with tantalum, range from about two- to eightfold, with binary tungsten-containing alloys exhibiting the best strengths at 1925 C (3500 F). The highest rupture strengths, 9,700 and 6,700 psi at 1 and 10 hours, respectively, were found for binary Ta-20W.

Tungsten and molybdenum additions are found to be about equally strengthening, on a weight per cent basis, in stress-rupture at 1480 and 1925 C (2700 and 3500 F). Thus, on an atomic per cent basis (i. e. , for more nearly equivalent low-temperature ductility and high-temperature tensile properties), alloys strengthened primarily with tungsten rather than molybdenum are more effective for time-dependent service applications. This is clearly demonstrated in the plots of 10-hour rupture strength versus atomic concentration at 1480 and 1925 C (2700 and 3500 F) shown in Figures 23 and 24, respectively.

For comparison, these figures also include values obtained for the dispersion alloy, Ta-5W-2.5Mo-0.5Zr-0.07C reported in the "Dispersion Effectiveness" section of this report. This alloy shows pronounced improvement in 10-hour rupture stress at 1480 C (2700 F) over an equivalent (about 11 atomic per cent total alloy addition) Ta-W alloy. At 1925 C (3500 F), the zirconium-carbon addition exhibits no significant effect on 10-hour rupture strength.

Recrystallization Behavior. Since a number of the molybdenum- and tungsten-containing alloys had been prepared for previous studies^(2,3) only those alloys that differed in composition or fabrication procedure were studied to assess their recrystallization performance. Recrystallization temperatures reported in Table 25 were based on results presented in Table 26.

Alloying with Group VI-A (Mo, W) metal additions continues to demonstrate significant effects in raising the recrystallization temperature of tantalum. As seen from Table 25, recrystallization temperatures are from 200 to about 800 C (360 to about 1440 F) higher than that of unalloyed tantalum.

Data of Table 25 for the molybdenum- and tungsten-plus-molybdenum-containing alloys show somewhat higher recrystallization temperatures than were expected, based on previously established parameters⁽³⁾. In most cases, these discrepancies can be traced to somewhat less cold work in materials of the current investigation, compared with the earlier materials studied.

Welding and Thermal Exposure. An evaluation of Ta-5Mo, Ta-12.5W, Ta-5W-2.5Mo, and Ta-10W-2.5Mo was conducted using automatic EB and TIG welding practices.

The rated capacity of the EB unit used 3 kva. EB welding conditions were:

| | |
|--------------|-------------------------|
| Beam voltage | 18 to 19 kilovolts |
| Beam current | 125 to 130 milliamperes |
| Travel speed | 3.5 ipm. |

TABLE 24. RUPTURE STRENGTHS OF TANTALUM AND TANTALUM-BASE ALLOYS AT 1925 C (3500 F)^(a)

| Alloy Composition (Balance Tantalum), weight per cent | Ultimate Tensile Strength, 1000 psi | Stress to Rupture ^(b) , 1000 psi, at Indicated Time | |
|---|--|--|---------|
| | | 1 Hour | 10 Hour |
| 100Ta | 3.9 ^(c) | 1.12 | 0.75 |
| 2.5Mo | -- | 3.8 | 2.58 |
| 5Mo | 9.0 ^(c) | 5.8 | 3.7 |
| 7.5Mo | -- | 6.4 | 4.1 |
| 5W | 7.1 ^(c) | 4.3 | 2.92 |
| 10W | 10.9 ^(c) | 7.05 | 4.8 |
| 20W | -- | 9.7 | 6.7 |
| 5W-2.5Mo | 9.0 ^(c) | 5.35 | 3.6 |
| 5W-5Mo | 12.9 ^(c) | 6.0 | 4.05 |
| 10W-2.5Mo | 12.5 ^(c) | 7.8 | 5.3 |

(a) Recrystallized material.

(b) Rupture strengths obtained graphically from a stress-rupture plot.

(c) Data from Reference 3.

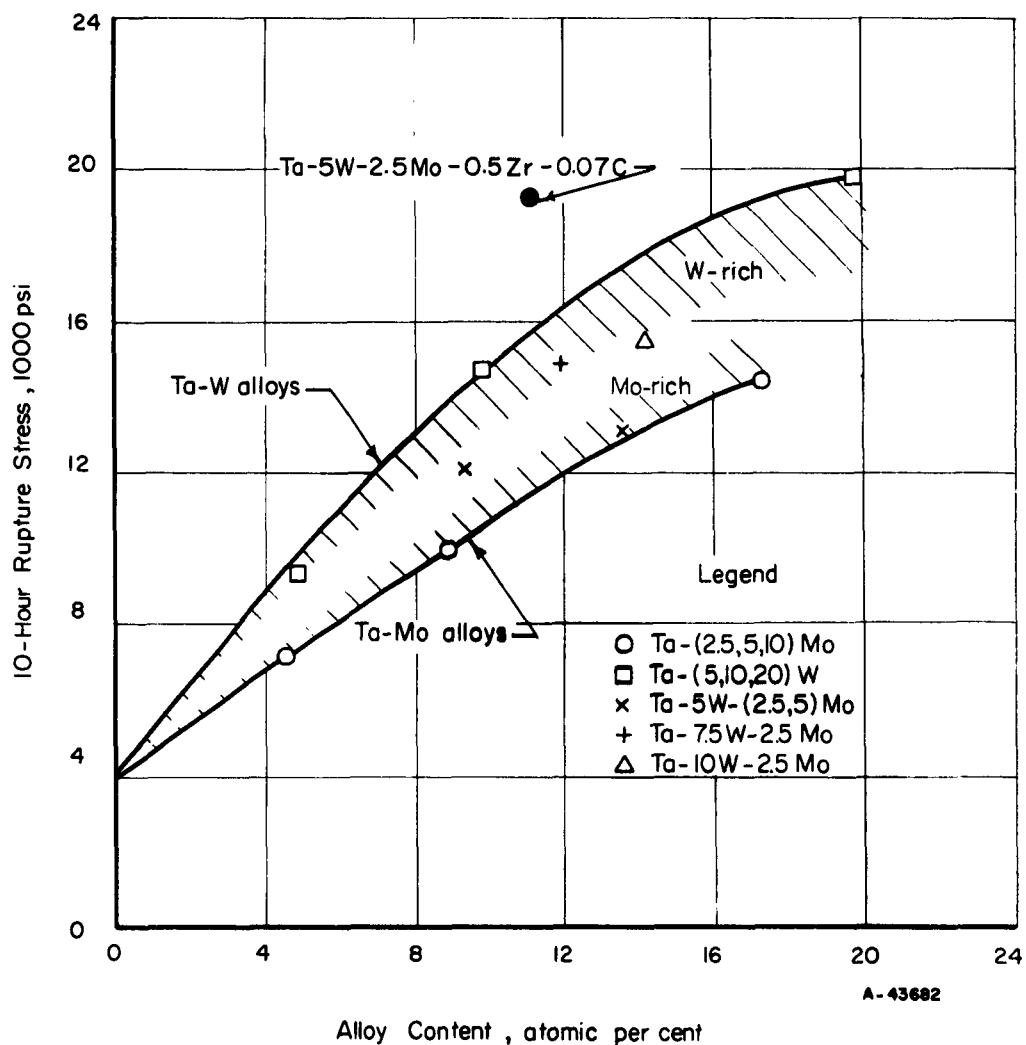


FIGURE 23. EFFECT OF MOLYBDENUM AND TUNGSTEN ADDITIONS ON THE RECRYSTALLIZED 10-HOUR RUPTURE STRESS OF TANTALUM AT 1480 C (2700 F)

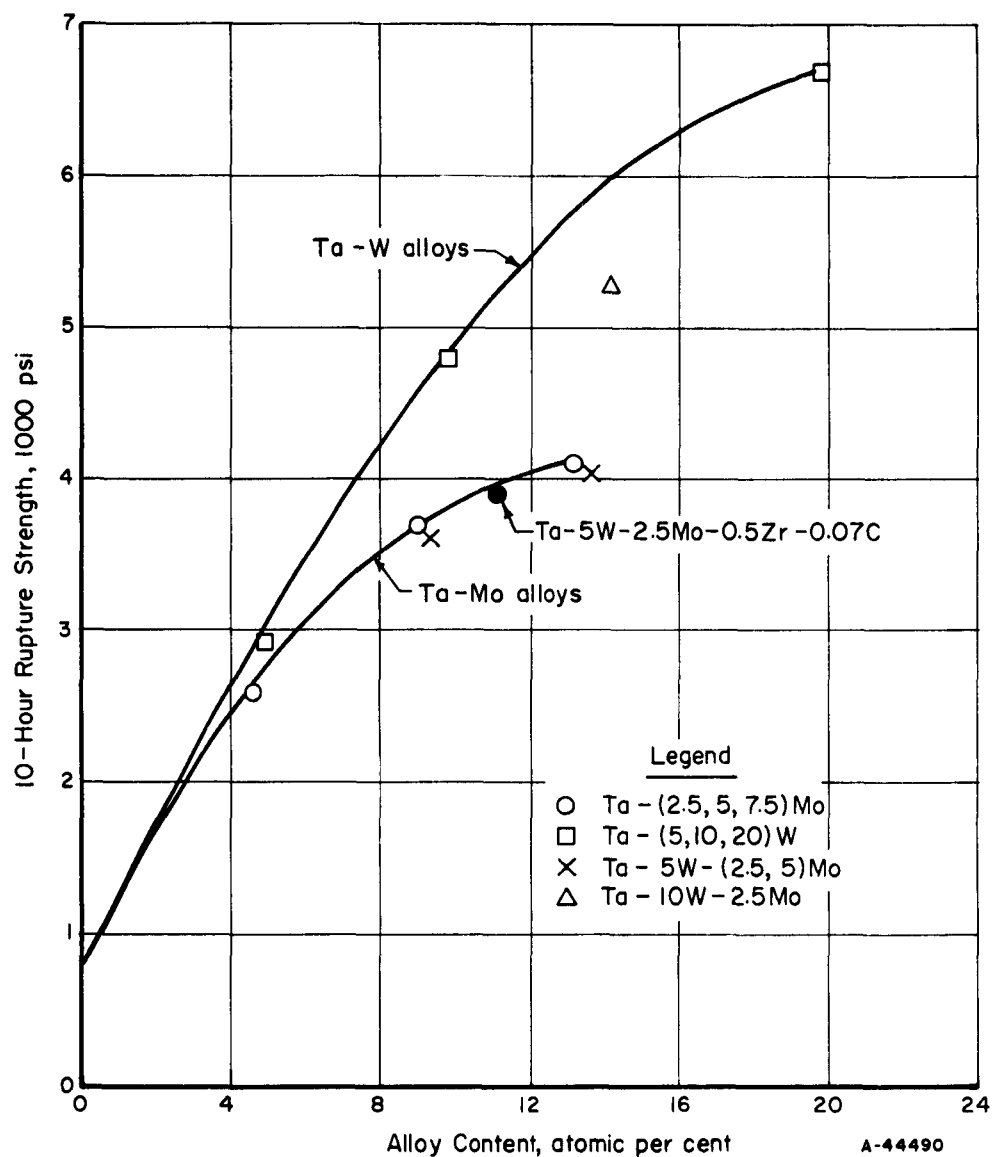


FIGURE 24. EFFECT OF MOLYBDENUM AND TUNGSTEN ADDITIONS ON THE RECRYSTALLIZED 10-HOUR RUPTURE STRESS OF TANTALUM AT 1925 C (3500 F)

TABLE 25. RECRYSTALLIZATION TEMPERATURES FOR SOLID-SOLUTION
TANTALUM-BASE ALLOYS

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Condition(a) | Recrystal- lization Temperature(b) | |
|---|-------------------|------------------------------------|--|--------|
| | | | C | F |
| 100TA(c) | -- | CR 65%/1 Hr 1200 C/CR 75% | 1200 | 2190 |
| 2.5Mo | 322A | WR 65% at 370 C/1 Hr 1400 C/CR 60% | 1400 | 2550 |
| 5W-7.5Mo | 331 | PR 75% at 1650 C | 1800 | 3270 |
| 5.2W-2.7Mo | 323 | WR 65% at 425 C/1 Hr 1400 C/CR 60% | 1600 | 2910 |
| 7.9W-4.1Mo | 324 | PR 75% at 1650 C | 1800 | 3270 |
| 10.6W-5.6Mo | 325 | PR 75% at 1650 C | (1900)(d) | (3450) |
| 15W-2.5Mo | 326A | PR 75% at 1650 C | (2000) | (3630) |

(a) CR = cold rolled; WR = warm rolled; PR = pack rolled.

(b) Complete recrystallization (75 per cent or greater) after 1-hour exposure in vacuum.

(c) Data from Reference 3.

(d) Values in parentheses are estimated.

TABLE 26. MICROSTRUCTURES AND HARDNESSES OF SOLID-SOLUTION TANTALUM-BASE ALLOYS^(a)

| Alloy Composition (Balance Tantalum), weight per cent | Specimen | Microstructure ^(b) and Hardness ^(c) , VHN, After Annealing 1 Hour at Indicated Temperature | | | | | | | | | | | | | | | | | | | | | | | |
|---|----------|--|---|---------|-----|----|-----|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|
| | | Cast, | | Wrought | | RT | | 1100 C (2010 F) | | 1200 C (2190 F) | | 1300 C (2370 F) | | 1400 C (2550 F) | | 1500 C (2730 F) | | 1600 C (2910 F) | | 1700 C (3090 F) | | 1800 C (3270 F) | | 1900 C (3450 F) | |
| | | RT | W | RT | W | RT | W | Rp | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R |
| 100Ta ^(d) | -- | -- | W | 134 | 100 | 80 | 85 | 85 | 85 | 85 | 85 | 85 | 85 | 85 | 85 | 85 | 85 | 85 | 85 | 85 | 85 | 85 | 85 | 85 | 85 |
| 2.5Mo | 322A | -- | W | 249 | 232 | Rb | Rp | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R | R |
| 5W-7.5Mo | 331 | -- | W | 405 | -- | -- | -- | W | 348 | 348 | 348 | 348 | 348 | 348 | 348 | 348 | 348 | 348 | 348 | 348 | 348 | 348 | 348 | 348 | 348 |
| 5.2W-2.7Mo | 323 | -- | W | 336 | 330 | W | 317 | 292 | 292 | 292 | 292 | 292 | 292 | 292 | 292 | 292 | 292 | 292 | 292 | 292 | 292 | 292 | 292 | 292 | 292 |
| 7.9W-4.1Mo | 324 | -- | W | 363 | -- | -- | -- | W | 316 ^(e) | 317 ^(f) | 317 ^(f) | 317 ^(f) | 317 ^(f) | 317 ^(f) | 317 ^(f) | 317 ^(f) | 317 ^(f) | 317 ^(f) | 317 ^(f) | 317 ^(f) | 317 ^(f) | 317 ^(f) | 317 ^(f) | 317 ^(f) | 317 ^(f) |
| 10.6W-5.6Mo | 325 | -- | W | 413 | -- | -- | -- | W | 363 | 363 | 363 | 363 | 363 | 363 | 363 | 363 | 363 | 363 | 363 | 363 | 363 | 363 | 363 | 363 | 363 |
| 15W-2.5Mo | 326A | -- | W | 455 | -- | -- | -- | W | 455 | 455 | 455 | 455 | 455 | 455 | 455 | 455 | 455 | 455 | 455 | 455 | 455 | 455 | 455 | 455 | 455 |

(a) Wrought condition given in Table 25.

(b) W = wrought

Rb = recrystallization beginning

Rp = recrystallization partially complete

R = recrystallization essentially complete.

(c) Minimum temperature for complete recrystallization (75 per cent or greater) is underlined.

(d) Hardness values are the average of five impressions using a 10-kg load.

(e) Data from Reference 3.

(f) 2-1/2-kg load.

(g) 5-kg load.

The beam current and voltage were close to the maximum obtainable with the unit. Consequently, any slight increase in beam voltage created an overload which shut off the unit. This made it extremely difficult to obtain a uniform bead along the length of the specimen. The slow travel speed that was necessary to obtain complete penetration in a single pass resulted in wider beads than desired and also heated the entire specimen to high temperatures. This heating caused distortion of the specimen halves, frequently resulting in mismatched weld joints.

The TIG welding was done in an inert gas-filled chamber. The chamber was pumped down to less than 1 micron prior to filling with helium. TIG welding conditions were:

| | |
|--------------|-------------------|
| Arc voltage | 15 to 19 volts |
| Arc current | 78 to 100 amperes |
| Travel speed | 7 ipm. |

Details of the automatic TIG welding conditions for specific alloys are given in Appendix III.

The results of examination of both EB and TIG welds of the four alloys for detailed evaluation, Ta-5Mo, Ta-12.5W, Ta-5W-2.5Mo, and Ta-10W-2.5Mo, are reported in Table 27. In general, the tungsten-arc welds had a much better appearance than the electron-beam welds. This can be attributed to the problems encountered with the low-voltage electron-beam unit. The slight fusion-line porosity noted in several of the welds was probably not enough to significantly affect weldment bend properties.

The photomicrographs presented in Figure 25 show base metal, heat-affected zone, and weld metal for the alloys welded by both EB and TIG techniques. Both types of welds show large grain sizes in the weld metal and a broad affected-structure zone. These characteristics are generally more common to TIG welds; the similar structure shown in this study for EB welds is attributed to difficulties encountered with the EB unit as explained earlier.

A more detailed examination of these welded alloy structures is recorded in the photomicrographs given in Figure 26. Because of better jigging and faster welding speeds in the TIG operation than in the EB operation, cooling rates were faster for TIG welds than for EB welds. This generally emphasized the cellular, cored structure of TIG welds, as shown in Figure 26, and also may have been responsible for finer grained structures occasionally observed in the heat-affected and weld zones of TIG welds.

Table 28 presents base-metal and weld-ductility data for the four alloys studied. Automatic welds exhibited ductility at room temperature in accord with their alloy contents; all alloys except the Ta-10W-2.5Mo composition displayed some weld ductility. This superior correlation displayed by the automatic TIG welds, compared with prior manual TIG weld-bend data, suggests superior control and reliability for the automatic process. Automatic EB welds exhibited somewhat better ductility at room temperature than the automatic TIG welds. The superior behavior of EB welds may reside in less contamination in this process (high vacuum versus "high purity" helium in the TIG process).

TABLE 27. RESULTS OF EXAMINATION OF AUTOMATIC EB- AND TIG-PRODUCED WELDS IN TANTALUM-BASE ALLOYS SELECTED FOR DETAILED EVALUATION

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Type Weld | Visual | Radiographic |
|---|-------------------|--------------|--|---|
| 5Mo | 99H | EB | Wide, uneven bead; pinholes in bead | Scattered fine pinholes or porosity |
| | | TIG | Good uniform penetration, bead slightly wider than desirable | Small amount of fine porosity along fusion line |
| 12. 5W | 266B | EB | Fairly even bead, slightly mismatched | No defects observed |
| | | TIG | Bead slightly wider than desirable, mismatched | Fine porosity along fusion line |
| 5W-2. 5Mo | 268D | EB | Width of bead uneven, mismatched at start and finish of weld | No defects observed |
| | | TIG | Bead wider than desirable, mismatched | No defects observed |
| 10W-2. 5Mo | 262E | EB | Nonuniform penetration, slightly mismatched | No defects observed |
| | | TIG | Bead wider than desirable, slightly mismatched | Small amount of fine porosity along fusion line |

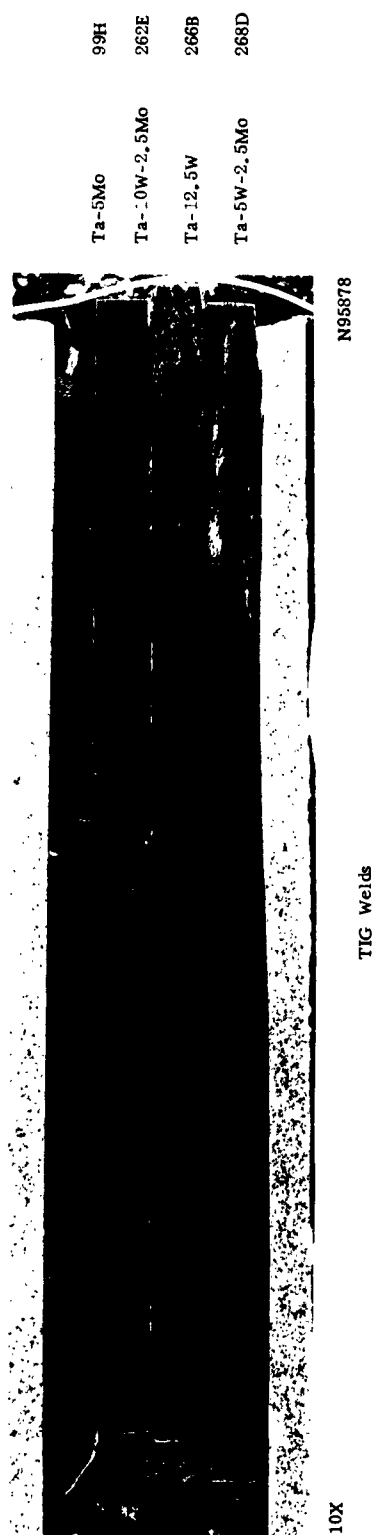
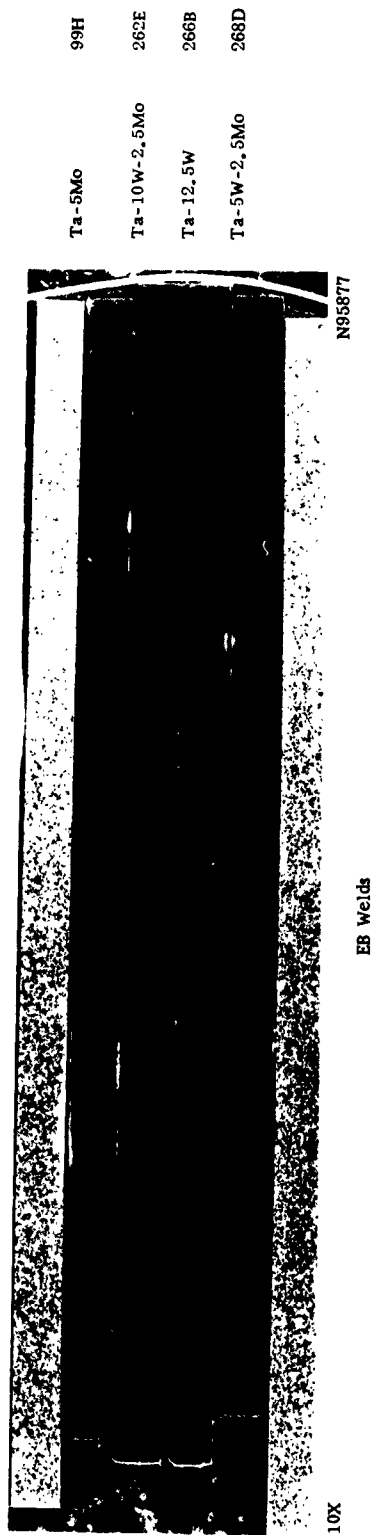
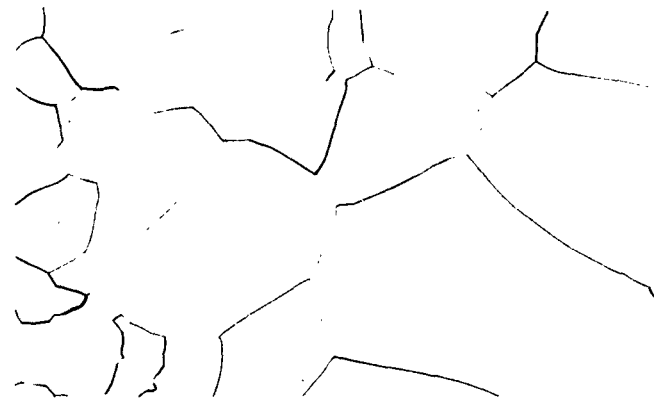


FIGURE 25. PHOTOMICROGRAPHS OF EB- AND TIG-WELDED SOLID-SOLUTION STRENGTHENED TANTALUM-BASE ALLOYS

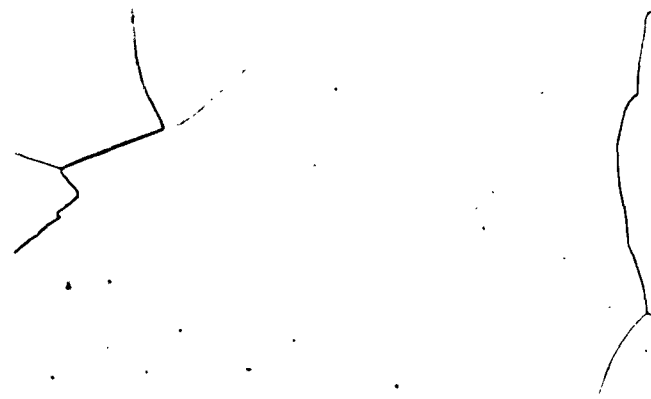
EB Welded - Ta-5Mo - 99H



Base



Heat-Affected Zone



Weld Metal

FIGURE 28. EFFECT OF EB AND TIG WELDING ON THE MICROSTRUCTURE OF SOLID-SOLUTION STRENGTHENED TANTALUM-BASE ALLOYS

TIG Welded - Ta-5Mo - 99H

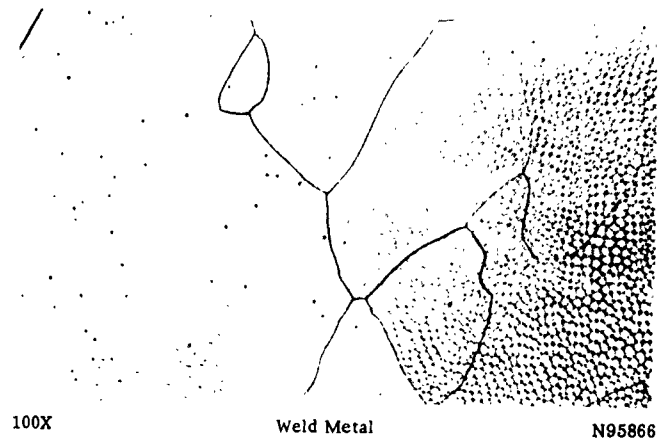
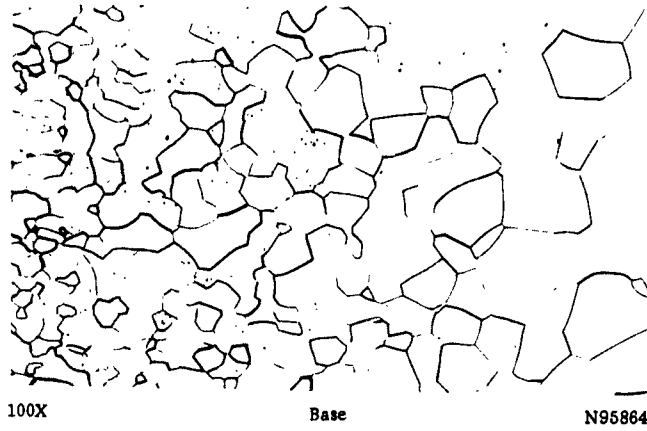


FIGURE 26. (CONTINUED)

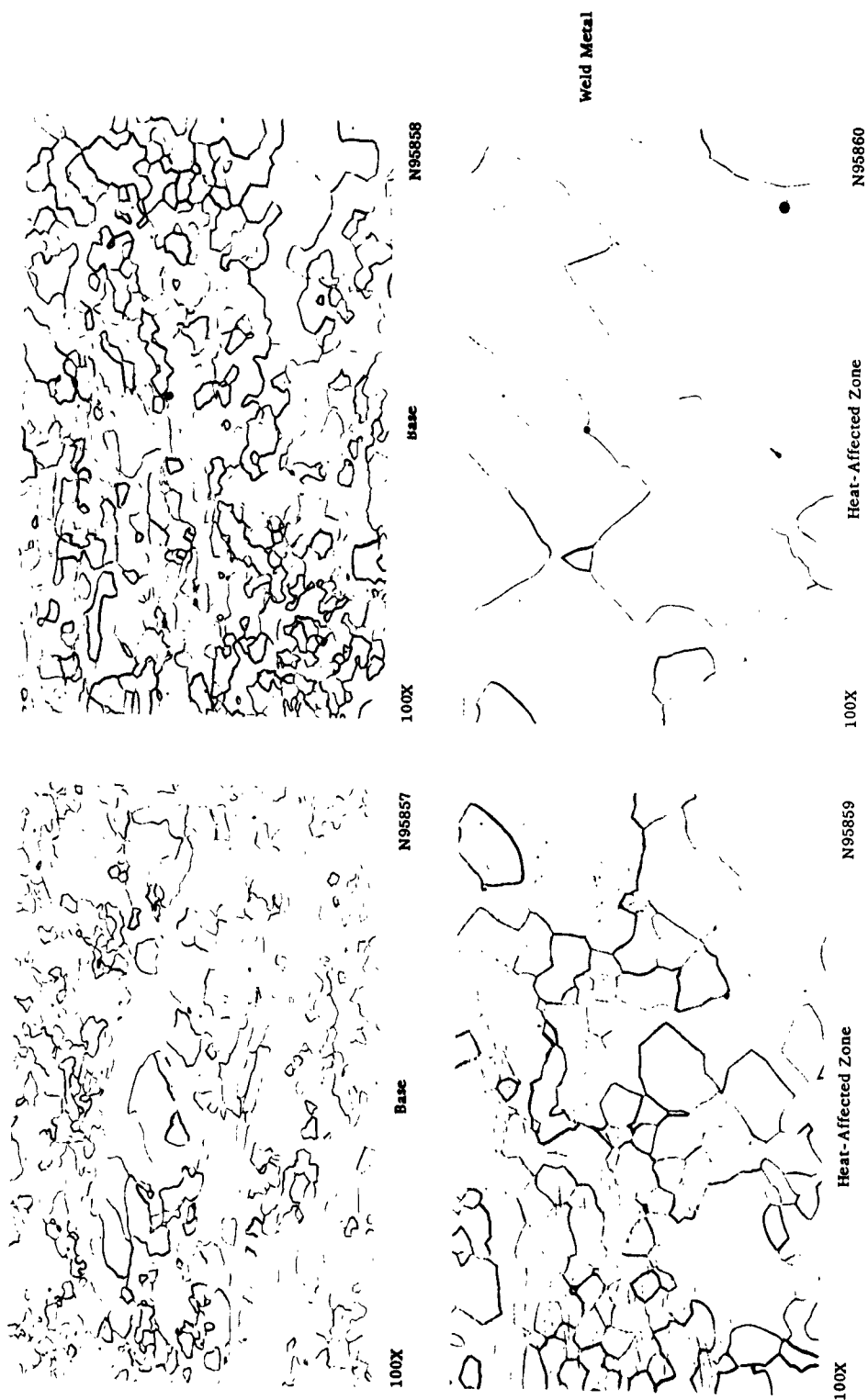


FIGURE 26 (CONTINUED)

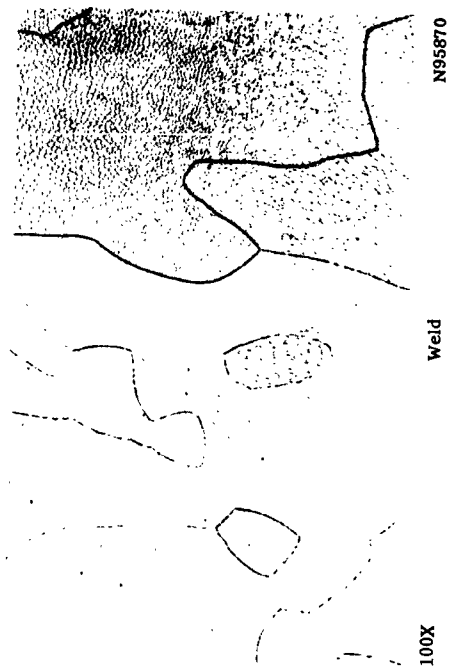
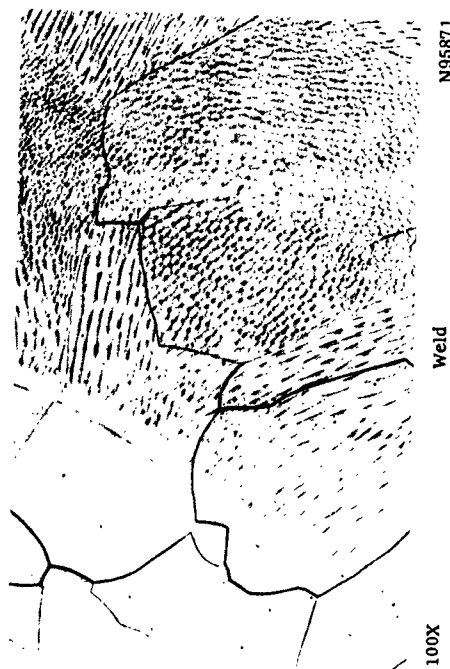
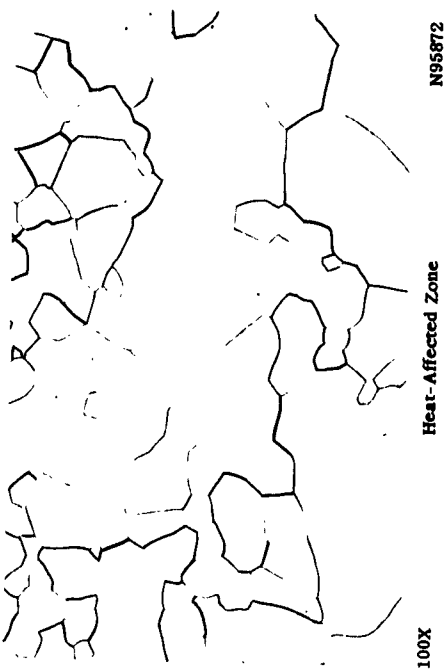
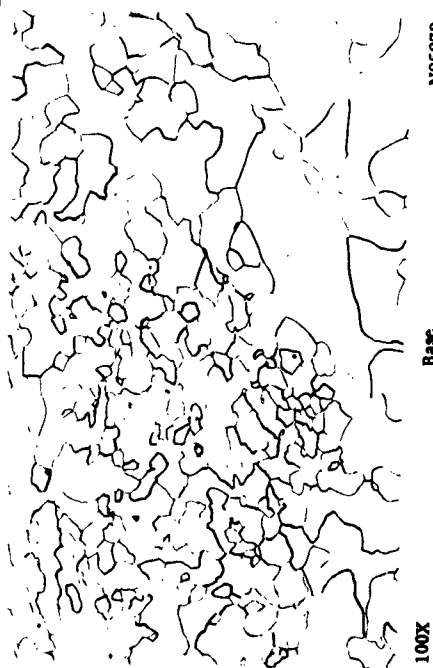


FIGURE 26. (CONTINUED)

EB Welded - Ta-5W-2.5Mo - 268D

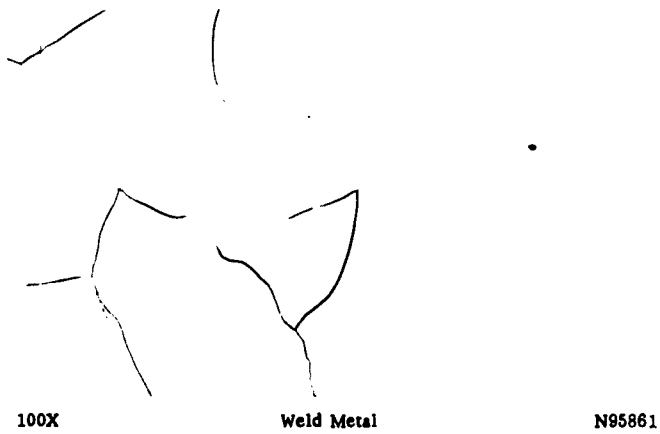
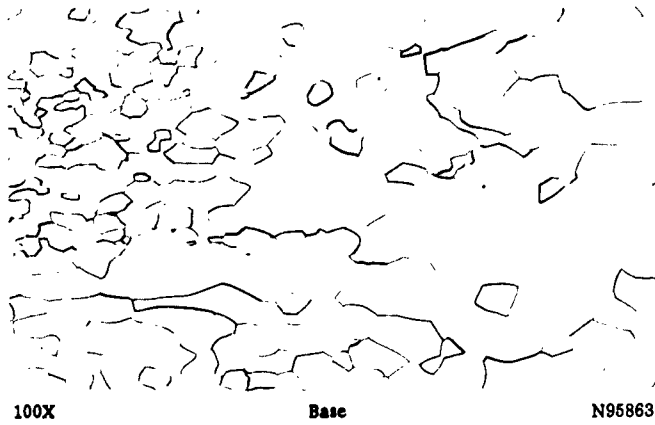


FIGURE 26. (CONTINUED)

TIG Welded - Ta-5W-2.5Mo - 268D

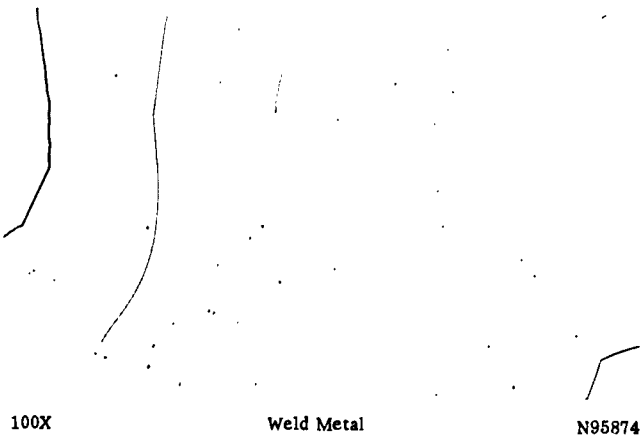
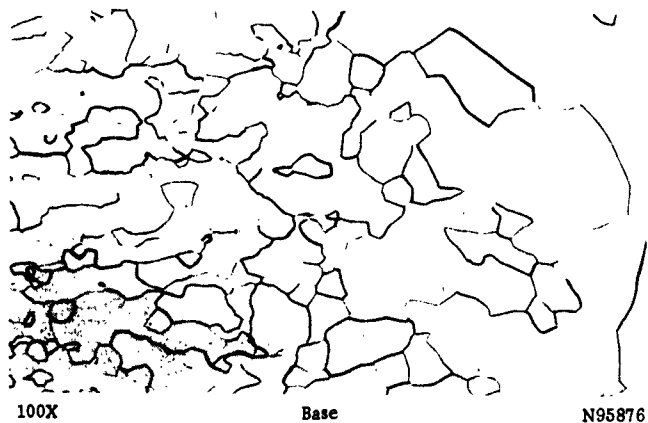


FIGURE 26. (CONTINUED)

EB Welded - Ta-10W-2.5Mo - 262E

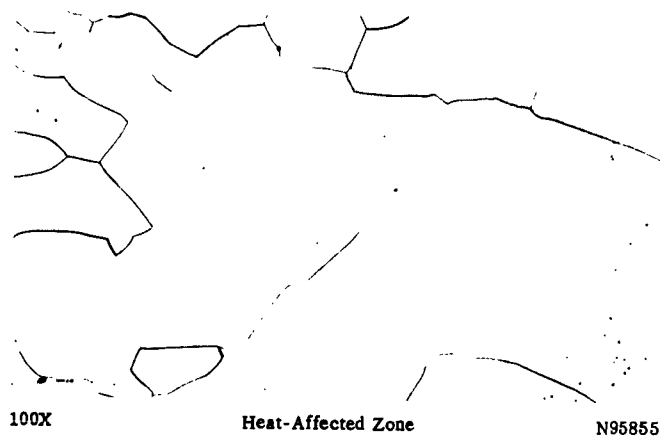
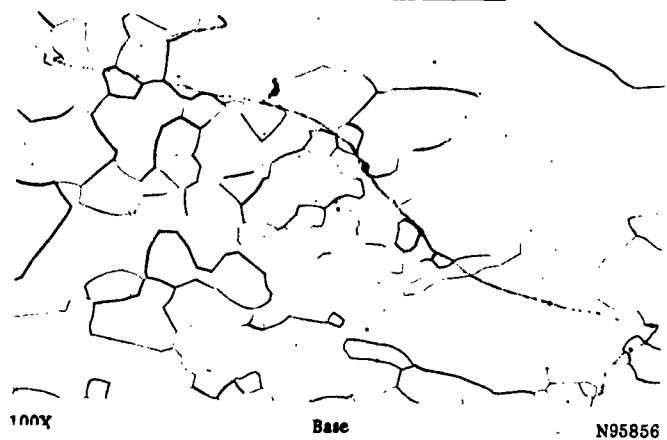


FIGURE 26. (CONTINUED)

TIG Welded - Ta-10W-2.5Mo - 282E

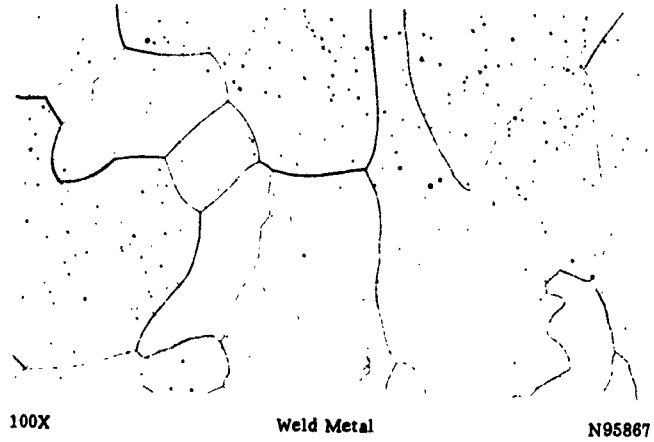
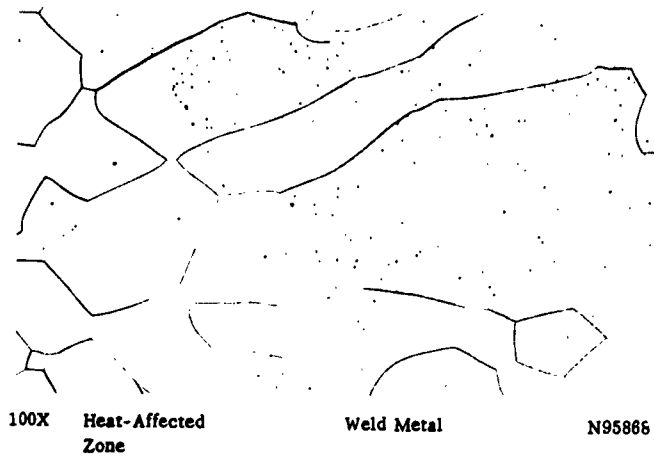
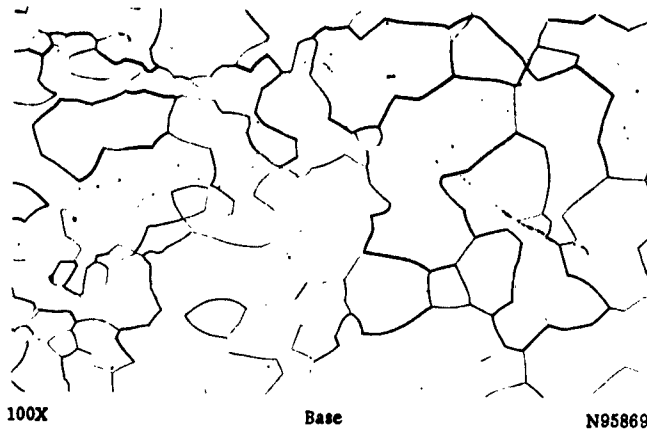


FIGURE 26. (CONTINUED)

TABLE 28. WELD-BEND DUCTILITIES OF TANTALUM-BASE ALLOYS(a)

| Alloy Composition (Balance Tantalum), weight per cent | Strengthening, atomic per cent | Alloy Specimen | Minimum-Bend-Radius Value, T(b) | | |
|---|--------------------------------------|-------------------|---------------------------------|------------------|----------------------------------|
| | | | Base Metal(c) | Manual TIG(c) | Automatic TIG Automatic EB |
| 5Mo | 9.0 | -- 99H | 0 | >22 | 5, 11, 14 3, 3, 6 |
| 12.5W | 12.3 | -- 266B | 0 | >22 | 22, 25 16, 16, 44 |
| 5W-2.5Mo | 9.4 | -- 268D | 0 | 2 | 6, 11, 12, 12 2, 6, 7 |
| 10W-2.5Mo | 14.2 | -- 262E | 1 | 19 | >41, >44 >42, 43 |

(a) Recrystallized material. Specimens bent perpendicular to the rolling direction and weld bead.

(b) T-value is radius of last good die before evidence of cracking appears, divided by specimen thickness.

(c) Data from Reference 3.

Although material welded by the EB welding process showed superior weld ductility to that welded by the TIG process, TIG welds were selected for further evaluation because (1) power limitations were experienced in the EB unit; (2) greater flexibility of the TIG unit; (3) greater room for improvement existed in weld-bend ductility of TIG welds; (4) more comparative data exist for TIG-welded refractory metal sheet than EB welded; and (5) MAB specifications are better defined for TIG welding.

The extended evaluation of TIG welding for Ta-5Mo, Ta-12.5W, Ta-5W-2.5Mo, and Ta-10W-2.5Mo included (1) room-temperature weld-bend ductility; (2) weld-bend transition behavior; (3) postweld thermal exposure; and (4) room-temperature weld tensile properties.

Welding conditions were similar to those used previously for TIG welding. Details are given in Appendix II for specific compositions. Table 29 presents visual and radiographic results. In general the quality of the welds was quite good (see Figure 27) with no porosity observed. This is an improvement over the previous TIG welds in the Ta-5Mo, Ta-12.5W, Ta-5W-2.5Mo, and Ta-10W-2.5Mo alloys.

A summary of the weld-bend ductilities of Ta-5Mo, Ta-12.5W, Ta-5W-2.5Mo, and Ta-10W-2.5Mo at various temperatures is given in Table 30. The as-welded bend ductility of both Ta-5Mo and Ta-5W-2.5Mo was good at 95 C (200 F), whereas 205 C (400 F) was required to exhibit good weld ductility for Ta-12.5W, and as high as 315 C (600 F) for Ta-10W-2.5Mo.

The data listed below suggest that welding these alloys causes an increase of about 320 to 455 C (575 to 820 F) in bend transition temperature.

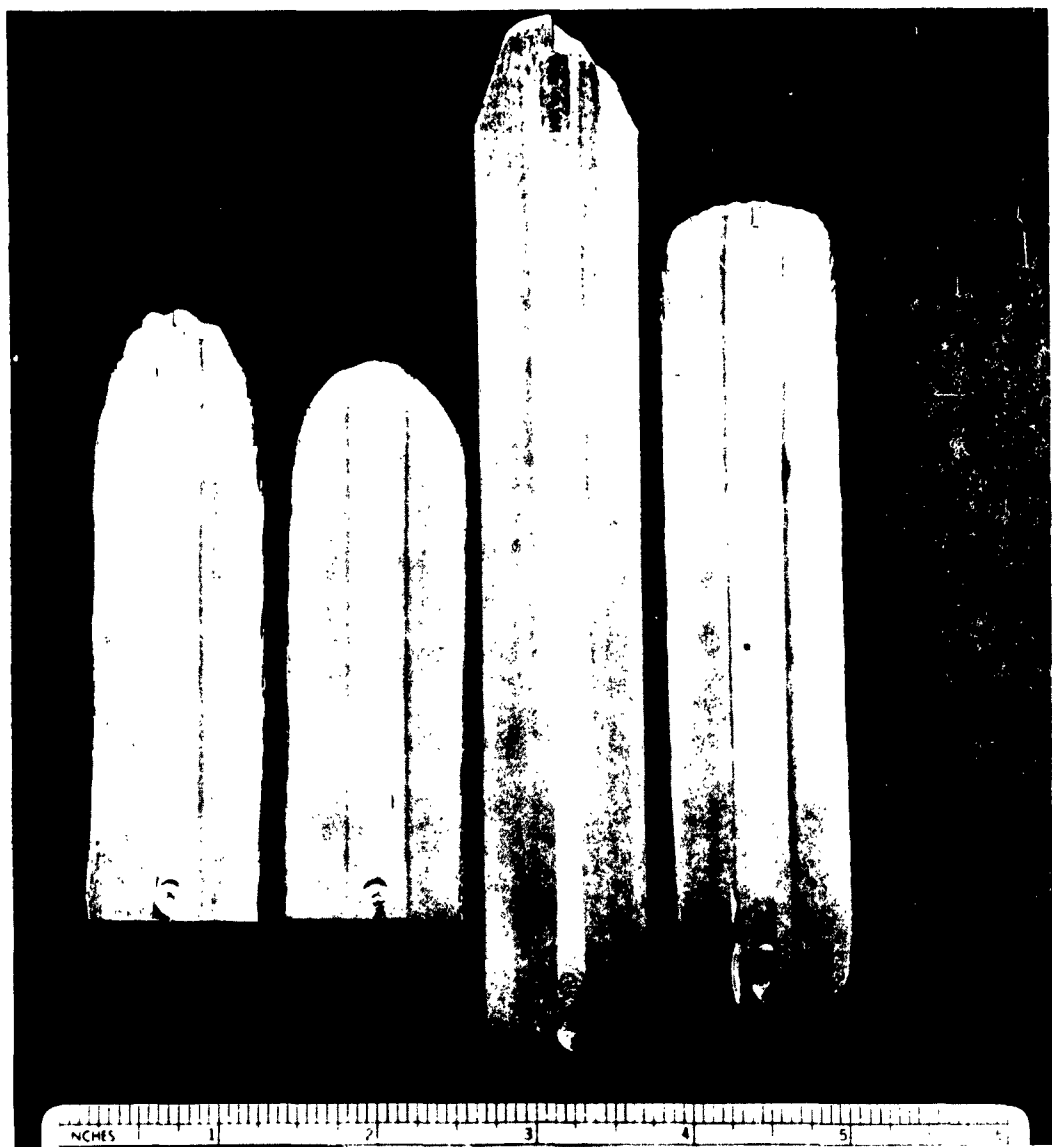
| Alloy | Approximate 4T Transition, C | | Increase in Temperature by Welding, C |
|--------------|------------------------------------|------|--|
| | Base(a) | Weld | |
| Ta-5Mo | < -250 | ~70 | ~ + 320 |
| Ta-12.5W | < -250 | 205 | ~ + 455 |
| Ta-5W-2.5Mo | < -250 | ~70 | ~ + 320 |
| Ta-10W-2.5Mo | -150 | ~290 | ~ + 440 |

(a) Approximated from Figure 16.

Also shown in Table 30 are the effects upon weld ductility of thermal exposures designed to simulate anticipated service conditions. The exposures included 1 and 10 hours at 1480 and 1925 C (2700 and 3500 F). After all but the 10-hour exposure at the highest temperature, the detrimental effects of welding upon ductility at room temperature were eradicated in all alloys. Room-temperature ductility of the weld metal was seriously degraded by the most extreme exposure, however. Improvements noted to result from the less severe exposures are believed to be attributable to homogenization of the cored welded structure. The degrading effects of the 10-hour 1925 C (3500 F) exposure were tentatively attributed to contamination during this most severe exposure.

TABLE 29. RESULTS OF EXAMINATION OF AUTOMATIC TIG-PRODUCED WELDS IN TANTALUM-BASE ALLOYS
SELECTED FOR DETAILED EVALUATION

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Visual | Radiographic |
|---|-------------------|---|--|
| 5Mo | 99I-1 | Very slightly mismatched, uneven penetration | No defects observed Ditto " " |
| | 99I-2 | Uneven penetration | |
| | 99J-1 | Good penetration, slightly mismatched | |
| | 99J-2 | Good penetration, slightly mismatched | |
| 12.5W | 266C-1 | Good penetration, penetration slightly more at ends than at center | " " " " |
| | 266C-2 | Good penetration, slightly mismatched | |
| | 266D-1 | Good penetration, slightly mismatched | |
| | 266D-2 | Good penetration, mismatched | |
| 5W-2.5Mo | 268E-1 | Good penetration | " " " " |
| | 268E-2 | Good penetration | |
| | 268H-1 | Penetration excessive at ends | |
| | 268H-2 | Penetration slightly excessive at ends, tapers to incomplete at center, possible contamination from hold-down bar at one spot | |
| 10W-2.5Mo | 262F | Mismatched, possible contamination from hold-down bar at one end | " " |
| | 262G | Mismatched, second pass not quite aligned on joint, resulting in uneven penetration | |



1X

Ta-5Mo
99J-1

Ta-12.5W
266C-2

Ta-5W-2.5Mo
268E-2

Ta-10W-2.5Mo
262F

N96352

FIGURE 27. PHOTOMACROGRAPH SHOWING TIG-WELDED TANTALUM-BASE ALLOY STRIPS PRIOR TO SURFACE GRINDING AND SAMPLE PREPARATION

TABLE 30. AUTOMATIC TIG WELD-BEND DUCTILITIES OF Ta-5Mo, Ta-12.5W, Ta-5W-2.5Mo, AND Ta-10W-2.5Mo (a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Condition | Minimum Bend Radius Value, T(b), at Indicated Temperature | | | |
|---|-------------------|-----------------------------------|--|--------------|---------------|---------------|
| | | | 25 C (75 F) | 95 C (200 F) | 205 C (400 F) | 315 C (600 F) |
| 5Mo | 99E | Base | 0,0,0,0 | -- | -- | -- |
| | 99H | As welded | 5,11,14 | -- | -- | -- |
| | 99I | As welded | -- | -- | 0 | -- |
| | 99J | As welded | 8,16 | 1,1 | 0 | -- |
| | 99I | As welded + 1 hr 1480 C (2700 F) | 0 | -- | -- | -- |
| | 99I | As welded + 10 hr 1480 C (2700 F) | 0 | -- | -- | -- |
| | 99I | As welded + 1 hr 1925 C (3500 F) | 0 | -- | -- | -- |
| | 99I | As welded + 10 hr 1925 C (3500 F) | 24 | -- | -- | -- |
| 12.5W | (c) | Base | 0 | -- | -- | -- |
| | 266B | As welded | 22,25 | -- | -- | -- |
| | 266C | As welded | -- | -- | 0 | -- |
| | 266D | As welded | -- | -- | 5 | -- |
| | 266C | As welded + 1 hr 1480 C (2700 F) | >25,>52 | 16,23 | -- | -- |
| | 266C | As welded + 10 hr 1480 C (2700 F) | 0 | -- | -- | -- |
| | 266C | As welded + 10 hr 1480 C (2700 F) | 0 | -- | -- | -- |
| | 266C | As welded + 1 hr 1925 C (3500 F) | 3 | -- | -- | -- |
| 5W-2.5Mo | 266C | As welded + 10 hr 1925 C (3500 F) | 44 | -- | -- | -- |
| | (c) | Base | 0 | -- | -- | -- |
| | 268D | As welded | 6,11,12,12 | -- | -- | -- |
| | 268E | As welded | -- | -- | 0 | -- |
| | 268H | As welded | 2,13 | 1,1,1 | 0,0 | -- |
| | 268E | As welded + 1 hr 1480 C (2700 F) | 0,0 | -- | -- | -- |
| | 268E | As welded + 10 hr 1480 C (2700 F) | 0,0 | -- | -- | -- |
| | 268E | As welded + 1 hr 1925 C (3500 F) | 0,0 | -- | -- | -- |
| 10W-2.5Mo | 268E | As welded + 10 hr 1925 C (3500 F) | 38,40 | -- | -- | -- |
| | (c) | Base | 1 | -- | -- | -- |
| | 262E | As welded | >41,>44 | -- | -- | -- |
| | 262G | As welded | -- | -- | 14,>24 | 0 |
| | 262F | As welded + 10 hr 1480 C (2700 F) | 2 | -- | -- | -- |
| | 262F | As welded + 10 hr 1925 C (3500 F) | >40 | -- | -- | -- |

(a) Recrystallized material.

(b) T-value is radius of last good die before evidence of cracking appears, divided by specimen thickness.

(c) Data from Reference 3.

For a complete evaluation of the tensile properties of welded materials, both longitudinal- and transverse-weld specimens should be used. In this program, the quantity of material available limited the evaluation to one type of specimen. The longitudinal-weld specimen was recommended because in this type specimen base metal, heat-affected zone, and weld metal are evaluated by equal strain on the entire section.

Table 31 presents room-temperature tensile properties of specimens of the selected alloys containing central welds along the tension axes. All alloys except Ta-10W-2.5Mo exhibited useful ductility at room temperature, although this was characteristically lower than base ductility. The ductilities agreed well with bend-test values, except for the most highly alloyed ductile material, Ta-12.5W, where bolstering of the weld metal by the surrounding base metal in the tension test was indicated. In no case was the weld zone observed to crack or otherwise fail prior to over-all specimen failure. Yield strength values increased progressively with increased alloying content, indicative of reliable, predictable behavior. Ultimate strength values reflected variations in both alloy content and strain hardening. In all cases, strengths were lower in the welded tensile specimen composites than for the base metal, although this was significant only for the Ta-12.5W alloy, the base value for which was obtained on fully recrystallized material. The severe 10-hour, 1925 C (3500 F) postweld exposure drastically reduced ductility in all alloys (except Ta-10W-2.5Mo, which was already brittle), but effects on the weld and surrounding base metal were equally severe as judged by these tests.

Effects of Rhenium and Ruthenium Additions

Past results⁽³⁾ have shown that simple binary additions of Groups VII-A (Re) or VIII-A (Ru, Os) metals to tantalum have significant effects on high-temperature tensile properties and recrystallization performance. These effects are greater than would be anticipated based on solid solution strengthening concepts alone. The additional boost in strengthening and recrystallization temperature is thought to be associated with an additional electronic contribution of these high-valency additions. Therefore, this phase of the program was selected to better understand and evaluate the effects of minor additions of rhenium and ruthenium to moderate-strength Ta-W and Ta-W-Mo alloys.

Fabrication

The four alloys selected for this evaluation were rolled 75 per cent at 1650 C (3000 F) directly to high-quality sheet. Data for these alloys are summarized in Table 32.

Evaluation

Evaluation of these rhenium- and ruthenium-containing tantalum-base alloys included 1925 C (3500 F) tensile properties, recrystallization behavior, low-temperature bend ductility, and manual TIG weld-bend tests. All material was recrystallized prior to testing.

TABLE 31. ROOM-TEMPERATURE TENSILE PROPERTIES OF AUTOMATIC TIG WELDS IN Ta-5Mo, Ta-12.5W, Ta-5W-2.5Mo, AND Ta-10W-2.5Mo(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Condition | Ultimate Tensile Strength, 1000 psi | Yield Strength, 0.2 Per Cent Offset, 1000 psi | Elongation in 1 Inch(b), per cent | Minimum Bend Radius Value, T(c) |
|---|-------------------|-----------------------------------|--|--|---|---------------------------------------|
| 5Mo | (d) | Base(e) | 141.4 | 139.0 | 10 | 0 |
| | 99J | As welded | 80.4 | 62.2 | 7 | 8, 16 |
| | 99I | As welded + 10 hr 1925 C (3500 F) | 98.8 | 95.5 | 3 | 24 |
| 12.5W | (d) | Base | 103.0 | 95.6 | 13 | 0 |
| | 266D | As welded | 90.7 | 84.8 | 6 | >25, >52 |
| | 266C | As welded + 10 hr 1925 C (3500 F) | 98.3 | -- | 2 | 44 |
| 5W-2.5Mo | (d) | Base(e) | 111.0 | 104.2 | 16 | 0 |
| | 268H | As welded | 90.7 | 74.4 | 10 | 2, 13 |
| | 268E | As welded + 10 hr 1925 C (3500 F) | 94.2 | 79.0 | 2 | 38, 40 |
| 10W-2.5Mo | (d) | Base | 102.8 | 91.0 | 25 | 1 |
| | 262G | As welded | 76.6 | -- | 1 | -- |
| | 262F | As welded + 10 hr 1925 C (3500 F) | 48.2 | -- | (1)(f) | >40 |

(a) Recrystallized material. Tested using conventional hydraulic loading and a crosshead speed of 0.01 inch per minute through 0.6 per cent offset yield strength, and 0.025 inch per minute to fracture (corresponding to approximate strain rates of 0.02 and 0.05 inch per inch per minute, respectively, for a 1/2-inch reduced section).

(b) 1/2-inch reduced section. Elongation measured from 1-inch gage marks in the shoulders.

(c) T-value is radius of last good die before evidence of cracking appears, divided by specimen thickness.

(d) Data from Reference 3.

(e) Wrought material.

(f) Failed outside reduced section in shoulders. Elongation estimated.

Bend Tests. Table 33 presents bend-ductility data for recrystallized tantalum-base alloys containing rhenium and ruthenium at 25 and -195 C (75 and -320 F). Three of the four alloys tested were room-temperature ductile. However, at -195 C (-320 F) only Ta-7.5W-2.5Re remained ductile.

Tensile Tests. Table 34 summarizes tensile properties determined for the evaluation of minor strengthening additions of rhenium and ruthenium to tantalum-base alloys at 1925 C (3500 F). Both rhenium and ruthenium were found to have potent hot-strengthening effects on Ta-W- and Ta-W-Mo-base alloys. Tensile strengths of these alloys range from about 25 to 55 per cent higher than laboratory-produced Ta-10W and up to about 70 per cent higher than commercially-produced Ta-10W. The strongest alloy tested was Ta-5W-5Re which had tensile and yield strengths of 16,900 and 16,300 psi, respectively. The four alloys containing rhenium or ruthenium exhibited some of the highest tensile and yield strengths at 1925 C (3500 F) yet achieved in room-temperature-ductile tantalum-base alloys. Particularly noteworthy is the outstanding combination of high- and low-temperature properties of Ta-7.5W-2.5Re - 14,800-psi tensile strength at 1925 C (3500 F) and 0T bend ductility at -195 C (-320 F).

Recrystallization Behavior. Table 35 summarizes the recrystallization temperature for the four rhenium- and ruthenium-containing alloys based on microstructural examination and hardness measurements, Table 36.

Auxiliary alloying of Ta-W and Ta-W-Mo bases with minor amounts of Groups VII-A (Re) and VIII-A (Ru) metal additions results in pronounced increase in the recrystallization temperature. In this regard, the unusually potent effect of singular additions of these metals defined in prior work⁽³⁾ was found to be additive to the effects of alloying with tungsten (and molybdenum).

Comparison of the effects of the various alloying additions of Groups VI-A (Mo, W), VII-A (Re), and VIII-A (Ru) metals is of interest. In addition to the level of alloying, electronic contribution of the various addition elements was expected to increase material properties, including the recrystallization parameters. Table 37 summarizes such effects. This comparison suggests that for maximum resistance to recrystallization, tungsten additions are favored over molybdenum; the Ta-15W-2.5Mo alloy resisted recrystallization to higher temperatures than did the Ta-10.6W-5.6Mo alloy, despite greater total additions (atomwise) in the latter alloy. Furthermore, the recrystallization temperatures of ruthenium- and rhenium-containing alloys were greater than would be expected based on atomic concentrations of additions, and agreed quite well with expectations based on considerations of electronic contribution. Mechanical-property data are insufficient to demonstrate a similar correlation of electronic alloying effects; intrinsic values for 1925 C (3500 F) tensile strength (Table 34) suggest the value of electronic contribution to strength, but other factors (i. e., lowering melting point, etc.) are undoubtedly of equal importance.

Welding. Results of visual and radiographic examination of manual TIG welds for tantalum alloys containing rhenium and ruthenium are given in Table 38. These alloys, welded by manual TIG techniques, were characterized by uneven and excessive penetration. The Ta-5W-2.5Ru cracked on welding which suggests a weld embrittling effect due to ruthenium.

TABLE 32. FABRICATION DATA FOR RHENIUM AND RUTHENIUM-CONTAINING TANTALUM-BASE ALLOYS

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Cast Hardness ^(a) , VHN | Rolling Tempera- ture ^(b) | | Final Reduction, per cent | Quality of Strip ^(c) |
|---|-------------------|--|--|------|---------------------------------|------------------------------------|
| | | | C | F | | |
| 5W-2.5Ru | 329 | 280 | 1650 | 3000 | 75 | Excellent |
| 5W-5Re | 328 | 304 | 1650 | 3000 | 75 | Excellent |
| 5W-2.5Mo-2.5Re | 330 | 278 | 1650 | 3000 | 75 | Excellent |
| 7.5W-2.5Re | 327 | 254 | 1650 | 3000 | 75 | Excellent |

(a) Hardness values are the average of five impressions using a 10-kg load.

(b) Alloys rolled in evacuated molybdenum packs.

(c) Excellent - no cracking of edges or surface.

TABLE 33. BEND DUCTILITIES OF RHENIUM AND RUTHENIUM-CONTAINING TANTALUM-BASE ALLOYS AT 25 AND -195 C (75 AND -320 F)(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Minimum Bend Radius Value, T ^(b) , at Indicated Temperature | |
|---|-------------------|---|----------------|
| | | 25 C (75 F) | -195 C (320 F) |
| 5W-2.5Ru | 329 | 5, 8 | 18, 18 |
| 5W-5Re | 328 | 2, 4 | >18, >18 |
| 5W-2.5Mo-2.5Re | 330 | 33, 33 | >16, >17 |
| 7.5W-2.5Re | 327 | 0, 0 | 0, 0 |

(a) Recrystallized material.

(b) T-value is radius of last good die before evidence of cracking appears, divided by specimen thickness.

TABLE 34. TENSILE PROPERTIES OF SOLID-SOLUTION-STRENGTHENED TANTALUM-BASE ALLOYS AT 1925 C (3500 F)(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Ultimate Tensile Strength, 1000 psi | Yield Strength, 0.2 Per Cent Offset, 1000 psi | Elongation in 1 inch per cent | Minimum Bend Radius Value, T(b), at Indicated Temperature 25 C (75 F) -195 C (-320 F) | Strength-to-Weight Ratio, 1000 psi/lb/in. ³ | |
|---|-------------------|--|--|-------------------------------------|---|---|-------------------|
| | | | | | | Tensile Strength | Yield Strength |
| 10W(c) | -- | 10.9 | 8.7 | 61 | 0 | 18.0 | 14.3 |
| 10W(d) | 10-4 | 10.0 | 8.3 | 53 | -- | 16.5 | 13.7 |
| 10W(e) | 10-2 | 10.0 | 10.0 | 58 | -- | 16.5 | 16.5 |
| 5W-2.5Ru | 329 | 13.9 | 12.8 | 13 | 5, 8 | 23.2 | 21.3 |
| 5W-5Re | 328 | 16.9 | 16.3 | 6 | 2, 4 | 27.7 | 26.7 |
| 5W-2.5Mo-2.5Re | 330 | 14.5 | 14.1 | (5)(f) | 33, 33 | 24.2 | 23.5 |
| 7.5W-2.5Re | 327 | 14.8 | 14.5 | 57 | 0, 0 | 24.2 | 23.7 |

- (a) Recrystallized material. Tested in vacuum using a mechanical screw-driven crosshead with a speed of 0.05 inch per minute for entire test unless otherwise noted (corresponding to an approximate strain rate of 0.04 inch per inch per minute - all specimens incorporated a 1-1/4-inch reduced section).
- (b) T-value is radius of last good die before evidence of cracking appears, divided by specimen thickness.
- (c) Data from Reference 3. Crosshead speed of 0.01 inch per minute up to the point of yielding and 0.05 inch per minute to fracture (corresponding to approximate strain rates of 0.008 and 0.04 inch per inch per minute, respectively, for a 1-1/4-inch reduced section).
- (d) Proof test on recrystallized commercial sheet. Crosshead speed as in (c) above.
- (e) Proof test on recrystallized commercial sheet. Crosshead speed as in (a) above.
- (f) Failed outside gage marks. Elongation estimated.

TABLE 35. RECRYSTALLIZATION TEMPERATURE FOR RHENIUM- AND RUTHENIUM-CONTAINING TANTALUM-BASE ALLOYS

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Condition(a) | Recrystallization Temperature ^(b) | |
|---|-------------------|---------------------------|---|------|
| | | | C | F |
| 100Ta ^(c) | -- | CR 65%/1 Hr 1200 C/CR 75% | 1200 | 2190 |
| 5W-2.5Ru | 329 | PR 75% at 1650 C | 1900 | 3450 |
| 5W-5Re | 328 | PR 75% at 1650 C | 1700 | 3090 |
| 5W-2.5Mo-2.5Re | 330 | PR 75% at 1650 C | 1800 | 3270 |
| 7.5W-2.5Re | 327 | PR 75% at 1650 C | 1700 | 3090 |

(a) CR = cold rolled

PR = pack rolled.

(b) Complete recrystallization (75 per cent or greater) after 1-hour exposure in vacuum.

(c) Data from Reference 3.

TABLE 36. MICROSTRUCTURES AND HARDNESSES OF RHENIUM- AND RUTHENIUM-CONTAINING TANTALUM-BASE ALLOYS(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Microstructures(b) and Hardness(c), VHN, After Annealing 1 Hour at Indicated Temperature | | | | | | | | | | | |
|---|-------------------|--|----|----------|-----|----------------|----------|----------|----------|----------------|----------------|----------------|----------|
| | | Cast, | | Wrought, | | 1100 C | | 1200 C | | 1300 C | | 1400 C | |
| | | RT | -- | RT | -- | (2010 F) | (2190 F) | (2370 F) | (2550 F) | (2730 F) | (2910 F) | (3090 F) | (3270 F) |
| 100Ta(d) | -- | -- | -- | W | 134 | R _p | W | R | 85 | R | 82 | -- | -- |
| | | | | | | 100 | 80 | | | | | | |
| 5W-2.5Ru | 329 | -- | 77 | W | 394 | W | W | W | W | R _b | R _p | R _p | R |
| | | | | | | 348 | 336 | 330 | 333 | 325 | 289 | 302 | 325 |
| 5W-5Re | 328 | -- | -- | W | 390 | W | W | W | W | R _b | R | R | R |
| | | | | | | 357 | 348 | 339 | 339 | 322 | 319 | 292 | 339 |
| 5W-2.5Mo-2.5Re | 330 | -- | -- | W | 366 | W | W | W | W | R _b | R _p | R _p | R |
| | | | | | | 325 | 322 | 319 | 319 | 325 | 292 | 299 | 351 |
| 7.5W-2.5Re | 327 | -- | -- | W | 339 | W | W | W | W | R _b | R | R | R |
| | | | | | | 309 | 306 | 306 | 294 | 281 | 247 | 258 | 299 |
| | | | | | | 254 | | | | | | | |

(a) Wrought condition given in Table 35.

(b) W = wrought

Rb = recrystallization beginning

R_p = recrystallization partially complete

R = recrystallization essentially complete.

Minimum temperature for complete recrystallization (75 per cent or greater) is underlined.

(c) Hardness values are the average of five impressions using a 10-kg load.

(d) Data from Reference 3.

TABLE 37. EFFECTS OF ADDITIONS TO TANTALUM UPON RECRYSTALLIZATION PROPERTIES

| Alloy Composition (Balance Tantalum), weight per cent | Total Additions, atomic per cent | Electronic Tungsten Equivalent(a), expressed as atomic per cent | Temperature to Complete Recrystallization in 1 Hour | |
|---|---|--|--|-------|
| | | | C | F |
| 2.5Mo | 4.6 | 4.6 | 1400 | 2550 |
| 5.2W-2.7Mo | 10 | 10 | 1600 | 2910 |
| 7.9W-4.1Mo | 15 | 15 | 1800 | 3270 |
| 5W-7.5Mo | 17.8 | 17.8 | 1800 | 3270 |
| 15W-2.5Mo | 18.9 | 18.9 | >1900 | >3450 |
| 10.6W-5.6Mo | 20 | 20 | >1800 | >3270 |
| 5W-2.5Ru | 9.2 | 18.0 | 1900 | 3450 |
| 7.5W-2.5Re | 9.8 | 12.2 | 1700 | 3090 |
| 5W-5Re | 9.8 | 14.7 | 1700 | 3090 |
| 5W-2.5Mo-2.5Re | 11.7 | 14.0 | 1800 | 3270 |

(a) Based on 6 electrons (s + d) per atom of tungsten, molybdenum

Based on 7 electrons (s + d) per atom of rhenium

Based on 8 electrons (s + d) per atom of ruthenium.

TABLE 38. RESULTS OF EXAMINATION OF MANUAL TIG-PRODUCED WELDS IN TANTALUM-BASE ALLOYS CONTAINING RHENIUM AND RUTHENIUM

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Visual | Radiographic |
|---|-------------------|---|---------------------|
| 5W-2.5Ru | 329 | Excessive penetration, cracked at one end, weld discolored | Confirmed crack |
| 5W-5Re | 328 | Excessive penetration | No defects observed |
| 5W-2.5Mo-2.5Re | 330 | Uneven penetration | Ditto |
| 7.5W-2.5Re | 327 | Uneven penetration | " |

Weld-ductility results for these screening alloys are given in Table 39. Alloys containing rhenium and ruthenium are seen to be brittle at room temperature in the as-welded condition.

DISCUSSION

This research program has investigated many of the facets critical to the exploitation of tantalum alloy technology. Tantalum alloys offer outstanding potential for structural use in the temperature range from about 1370 to 2205 C (2550 to 4000 F), where attractive hot strength is coupled with ease of fabricability, insensitivity to man-handling at ambient temperatures, weldability, etc.

The effects of the hot-strengthening major additions of tungsten and molybdenum upon such critical alloy behavior have been assessed in the present program. Tungsten was found to be somewhat less degrading to the low-temperature behavior of tantalum than molybdenum, and at the same time tungsten promotes at least equal hot strengthening to that of molybdenum, its closest hot-strengthening rival. For time-dependent service at elevated temperatures, tungsten additions are definitely superior to molybdenum, even when considered on a strength/density basis. Thus, tungsten has been defined as the most effective major alloying element for tantalum. Parameters established in this program indicate that tantalum can tolerate up to about 13 per cent tungsten without serious degradation of ductility at liquid-hydrogen temperature, or up to about 19 per cent tungsten without serious degradation of ductility at room temperature. From current and past studies, expected properties of selected binary alloys, obtained by interpolation and limited extrapolation of established parameters, are tabulated below:

| Alloy | 4T Bend Transition Temperature | 1480 C (2700 F) | | 1925 C (3500 F) | |
|--------|--------------------------------------|----------------------------------|----------------------------------|----------------------------------|----------------------------------|
| | | Strength | | Strength | |
| | | Tensile Strength, 1000 psi | 10-Hour Strength, 1000 psi | Tensile Strength, 1000 psi | 10-Hour Strength, 1000 psi |
| Ta | <-250 C (<-420 F) | 6 | 4 | 4 | 0.8 |
| Ta-13W | -250 C (-420 F) | 34 | 17 | 13.2 | 5.7 |
| Ta-14W | -195 C (-320 F) | 36 | 17.8 | 14.0 | 5.9 |
| Ta-19W | 25 C (75 F) | 45 | 19.6 | 17.5 | 6.6 |

Although such high-strength solid solution strengthened alloys are readily fabricable by appropriate techniques, degradation of ductility as a result of welding may severely restrict their engineering utilization. Preliminary studies suggested that improved welding techniques will be required to achieve useful weld ductility at room temperature in alloys containing more than about 13 to 14 per cent tungsten.

Auxiliary additions of high-valence elements such as rhenium or ruthenium markedly improve hot strength, and are far more effective than additional tungsten, atom for atom, in this regard. However, such additions are also far more degrading

TABLE 39. MANUAL TIG WELD-BEND DUCTILITIES OF TANTALUM-BASE ALLOYS CONTAINING RHENIUM AND RUTHENIUM^(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Condition | Minimum Bend Radius Value, T ^(b) , at Room Temperature |
|---|-------------------|-----------|--|
| 5W-2.5Ru | 329 | Base | 5, 8 |
| | 329 | As welded | (c) |
| 5W-5Re | 328 | Base | 2, 4 |
| | 328 | As welded | >40 |
| 5W-2.5Mo-2.5Re | 330 | Base | 33, 33 |
| | 330 | As welded | (d) |
| 7.5W-2.5Re | 327 | Base | 0, 0 |
| | 327 | As welded | >39 |

(a) Recrystallized material.

(b) T-value is radius of last good die before evidence of cracking appears, divided by specimen thickness.

(c) Cracked on welding.

(d) Cracked on grinding.

to the low-temperature behavior of tantalum-base alloys than are tungsten additions. Considering combined properties, the current work showed that these exotic additions are no more beneficial than tungsten, as shown by the following tabulation of properties (values for hypothetical Ta-W alloys were obtained by interpolation from established values):

| Alloy | Alloying Level | | 4T Bend Transition Temperature | Tensile Strength at 1925 C (3500 F), 1000 psi |
|-------------------|--------------------|--|--------------------------------------|---|
| | Atomic Per Cent | Electronic W Equivalent, atomic per cent | | |
| Ta-7.5W-2.5Re | 9.8 | 12.2 | <-195 C (<-320 F) | 14.8 |
| Ta-15.3W | 15 | 15 | -160 C (-260 F) | 14.8 |
| Ta-14.3W | 14 | 14 | -200 C (-330 F) | 14.0 |
| Ta-5W-2.5Mo-2.5Re | 11.7 | 14.0 | >25 C (>75 F) | 14.5 |
| Ta-9W-4.6Mo | 17 | 17 | 10 C (50 F) | 14.5 |
| Ta-5W-5Re | 9.8 | 14.7 | 25 C (75 F) | 16.9 |
| Ta-18.3W | 18 | 18 | -10 C (15 F) | 16.9 |
| Ta-5W-2.5Ru | 9.2 | 18.0 | 50 C (120 F) | 13.9 |
| Ta-14W | 13.7 | 13.7 | -210 C (-345 F) | 13.9 |

Thus, in one case (Ta-7.5W-2.5Re), the effects of rhenium additions were slightly superior to the expected values for tungsten, and in two cases, it was slightly inferior in its effects upon combined high- and low-temperature properties. In all cases differences were within the limits of experimental error. Auxiliary ruthenium additions are seen to be markedly inferior to simple tungsten additions. No weldability advantages were found for the alloys containing the exotic additions. (Weld ductility of the Ta-7.5W-2.5Re alloy was nil at room temperature, a result not expected in view of its excellent cryogenic base ductility.) Clearly, these comparisons, which are based on established Ta-W alloy parameters, do not support the use of high-valence solid solution strengtheners.

The effects of additions designed to promote dispersed-phase strengthening of the Ta-5W-2.5Mo base composition were investigated. Both simple (but not well understood) reactive metal and reactive-metal-carbide dispersoids were studied. Reactive metal additions were observed to exhibit pronounced strengthening in tensile tests at 1925 C (3500 F), the only strength evaluation conducted, so that, for example, 1 atomic per cent of zirconium appeared equivalent to about 4 to 4.5 atomic per cent tungsten in 1925 C (3500 F) strengthening capacity. At low temperatures, reactive (Zr, Hf) metal additions were somewhat degrading to ductility, so that 1 atomic per cent zirconium exhibited about the same effect as 2 or 2.5 atomic per cent molybdenum or tungsten, respectively. In other words, a zirconium-free Ta-W-Mo alloy (with 1:1 atomic W:Mo ratio) would need 18 atomic per cent combined W + Mo, i. e., a Ta-9.5W-5Mo alloy, to equal the 15,100-psi strength exhibited by the Ta-5W-2.5Mo-1Zr (2 atomic per cent zirconium) alloy at 1925 C (3500 F). Such an alloy would exhibit a ductile-brittle transition temperature of about 60 C (140 F) compared to the -195 C (-320 F) value measured for the zirconium-containing variety. The optimum level for auxiliary reactive metal strengthening at 1925 C (3500 F) appeared to be about 1 to 2 atomic per cent.

Carbide dispersions were not nearly so effective as the simple reactive metal additions; tensile strengthening at 1925 C (3500 F) was inferior, and degradation of low-temperature behavior was probably somewhat more severe. However, the carbide-containing Ta-5W-2.5Mo-0.5Zr-0.07C alloy exhibited attractive short- and long-time strengthening advantages over the Ta-5W-2.5Mo base composition at 1480 C (2700 F). Thus, at 1480 C (2700 F), "ZrC" additions appeared about as effective, considering both high- and low-temperature behavior, with respect to simple tungsten additions as the carbon-free variety did at 1925 C (3500 F). Weldability comparisons cannot be made, as the carbide-dispersion alloy welds were brittle at room temperature, and transition behavior was not determined. Excellent retention of strength of this alloy in the recrystallized condition [recrystallized carbide-containing material was stronger than fibered material at 1480 C (2700 F)] and the demonstrated importance of process annealing temperature supports Chang's findings⁽¹⁴⁾ regarding strain-induced precipitation in similar columbium alloys.

The fabrication parameter that significantly improved hot strength in the Ta-5W-2.5Mo-0.5Zr-0.07C alloy, solution annealing, also seriously degraded subsequent fabrication, making utilization of this finding practically untenable in the current state of development of production technology. Carbon-free alloys have greater freedom in this regard, although the importance of high-temperature process annealing to properties of the carbon-free alloys was not evaluated.

Figure 28 illustrates the hot-strength spectrum of attractive tantalum-base alloys, and shows in particular strengths of the more promising alloys of the current investigations. Alloys whose strengths are represented by the lower regions of the band shown in Figure 28 generally possess excellent cryogenic ductility; however, this degenerates as the strength level is raised at any given temperature.

Figure 29 presents a more rigorous comparison of high- and low-temperature behavior of several promising tantalum-base alloys. From this presentation, three of the alloys investigated appear superior to binary Ta-W alloys:

Ta-5W-2.5Mo-1Zr

Ta-7.5W-2.5Re

Ta-5W-2.5Mo-1Hf.

Considering that one-half of the solid solution strengthener in the Ta-W-Mo-(reactive metal) alloys was molybdenum, which carries with it definite degrading characteristics relative to the expected effects of tungsten, the effects of zirconium and hafnium additions are pronounced indeed. As described in the text, the improvement by the minor rhenium addition in the Ta-7.5W-2.5Re alloy is open to question.

Although the strength and ductility values and parametric behavior of tantalum-base alloys apply specifically to the conditions of alloy purity, fabrication history, and testing procedures utilized in this experimental program, they are believed more than mere qualitatively valid descriptors of tantalum alloy technology. For example, it is quite certain that tungsten additions are substantially superior to molybdenum additions regarding stress-rupture durability and transition behavior, that at least 16 to 17 per cent tungsten can be tolerated by tantalum without serious embrittlement at room temperature (with "good" commercial production practice), that minor zirconium additions

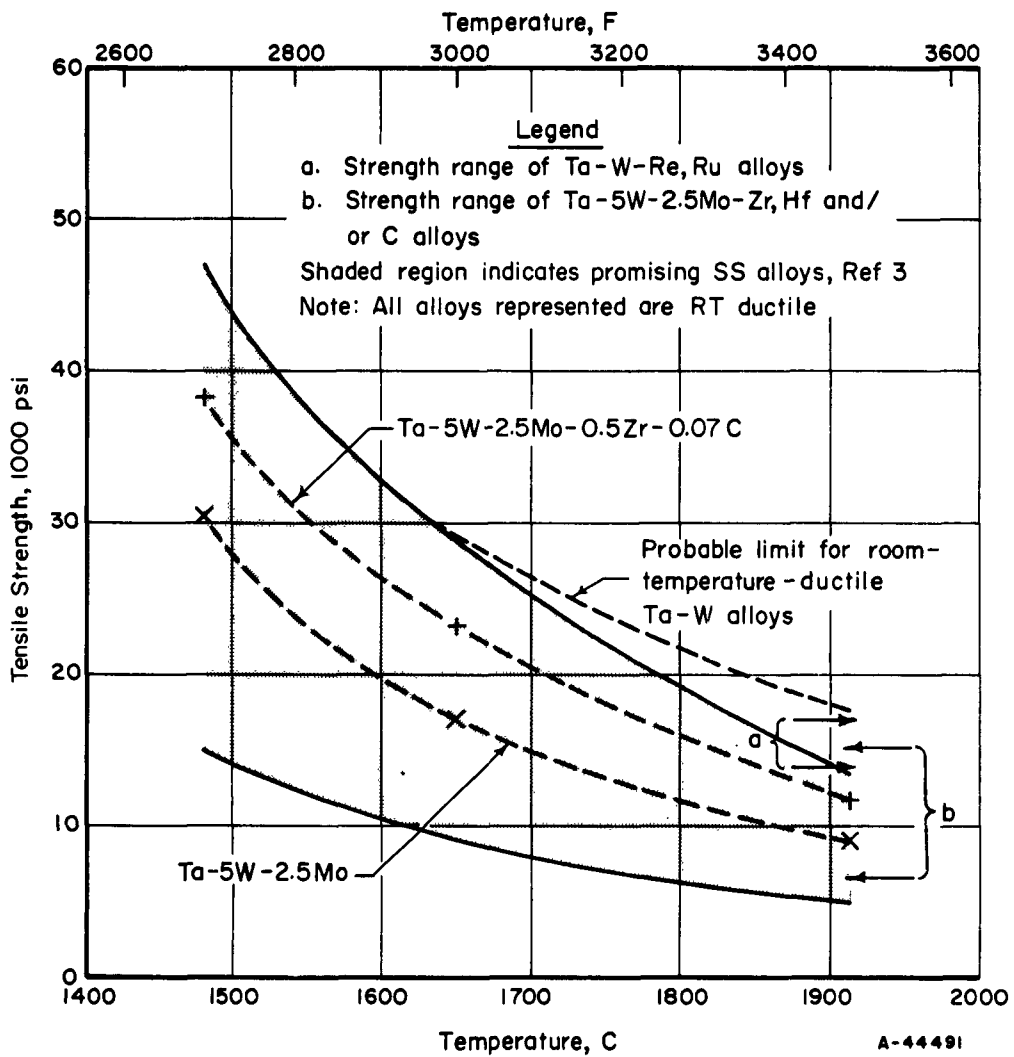


FIGURE 28. EFFECT OF TEMPERATURE ON THE RECRYSTALLIZED TENSILE STRENGTH OF TANTALUM-BASE ALLOYS

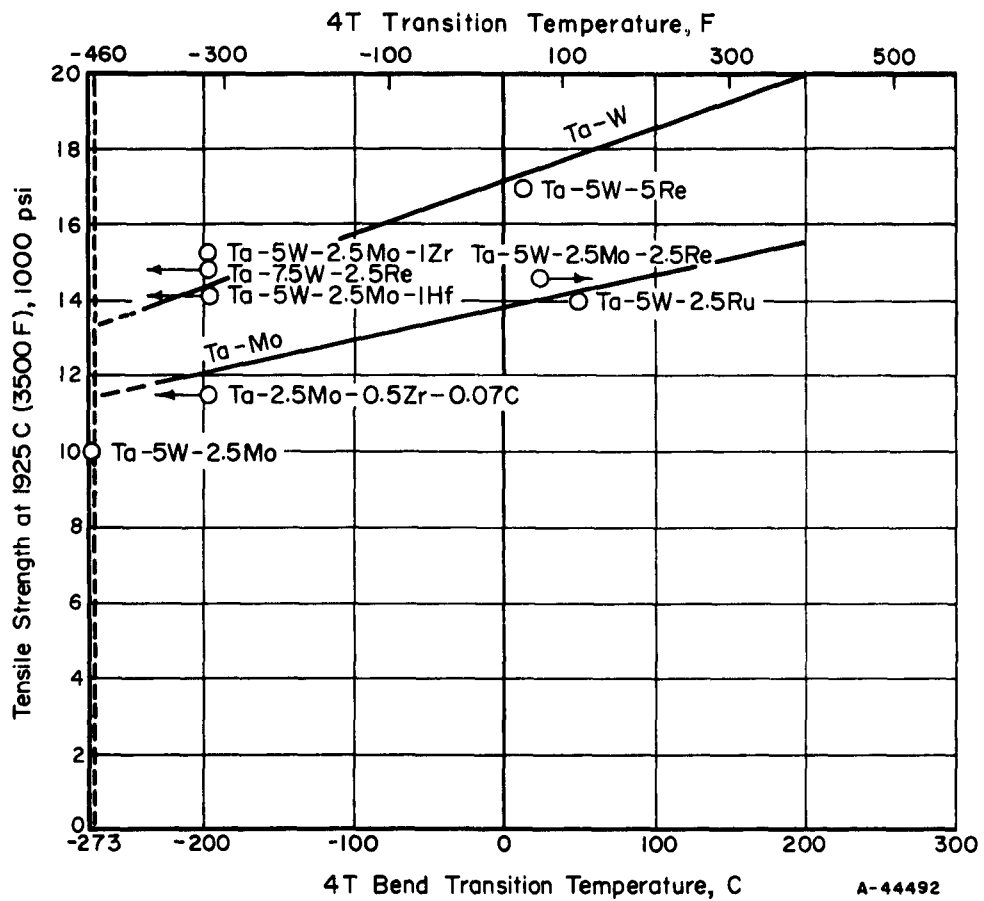


FIGURE 29. ATTAINABLE STRENGTH AT 1925 C (3500 F) AS A FUNCTION OF TRANSITION TEMPERATURE

are superior to "ZrC" additions regarding maximum temperature capability for useful strengthening, and that rhenium additions are not outstandingly beneficial to over-all alloy performance.

CONCLUSIONS

Based on the data presented in this report, the following major conclusions are justified:

- (1) Auxiliary additions of zirconium or hafnium to tungsten- or molybdenum-containing tantalum-base alloys are very effective strengtheners at 1925 C (3500 F). Effects on low-temperature behavior are relatively mild. Ta-W alloys containing about 1 weight per cent zirconium possess potential for the most attractive combination of hot [1925 C (3500 F)] strength and cold ductility of known tantalum-base alloys.
- (2) In comparison with zirconium (or hafnium), carbide dispersions are ineffective strengtheners at 1925 C (3500 F), and probably are more detrimental to low-temperature ductility. Carbide dispersions cannot be used effectively at 1925 C (3500 F). At 1480 C (2700 F) "ZrC"-type dispersions are effective short- and long-time strengtheners; for service at this temperature, the combined hot-strength-cold-ductility behavior of such alloys is superior to the Ta-W class of alloy. Maximum strength in the Ta-(W/Mo)-("ZrC") class of alloy at 1480 C (2700 F), obtained through solution process annealing, is currently untenable in the practical sense because of concomitant degradation of fabricability and probably low-temperature ductility.
- (3) At 1480 and 1925 C (2700 and 3500 F) tungsten additions are markedly superior to molybdenum additions on an atomic per cent basis for time-dependent applications. Suspected W-Mo interaction strengthening in Ta-W-Mo alloys, indicated by hot tensile strengths in prior research, did not materialize in stress-rupture tests in the current effort. More severe degradation of cryogenic ductility by molybdenum additions compared with tungsten additions on an atomic per cent basis further detracts from the utility of molybdenum additions. Thus, the use of molybdenum as an alloying element for strengthening tantalum at high temperatures cannot be justified on the basis of the current research program.
- (4) High-valence additions (rhenium and ruthenium in the current studies) are markedly effective hot strengtheners, more so than tungsten on an atomic basis. However, this advantage is nullified by concomitant severe degradation to low-temperature ductility. Use of these exotic strengtheners is probably not justified, although data for Ta-7.5W-2.5Re warrants further investigation of this type of alloy.

- (5) Weldability (weld ductility) is the major problem restricting the mechanical utility of high-strength tantalum-base alloys. Increases in ductile-to-brittle transition temperature of from 300 to 500 C (540 to 900 F) probably result primarily from coring in the weld metal. Some hope for at least partial amelioration of this effect by improved welding practice is suggested by the recovery of weld ductility resulting from postweld thermal exposure [e.g., 1 hour at 1480 C (2700 F)] of solid solution strengthened alloys.

RECOMMENDATIONS

Several areas for additional study are suggested by the results of this research program. A number of these have already been implemented by ASD.

- (1) Conduct detailed investigations of production capabilities for, and mechanical behavior (broad exploration of low- and high-temperature properties and effects of fabrication on properties) of three classes of alloys:
 - (a) Ta-(15-20)W
 - (b) Ta-(10-15)W-0.5Zr-0.07C
 - (c) Ta-(10-15)W-1Zr.
- (2) To augment the effectiveness of auxiliary reactive metal additions, study their interaction with dispersion-forming interstitial additions other than carbon [carbides are unstable at temperatures approaching 1925 C (3500 F)].
- (3) Repeat and expand investigations of Ta-7.5W-2.5Re and similar alloys containing from about 7.5 to 12.5 per cent tungsten in combination with 1 to 4 per cent rhenium, with and without 1 per cent zirconium.
- (4) Initiate a comprehensive welding process development program for high-strength tantalum-base alloys. Both pressure- and fusion-welding techniques should be considered. For fusion welding, rapid weld-traverse speeds, effective jig cooling, and the possibility of repeat, subfusion passes for postweld structural homogenization should be emphasized.

* * * * *

Data upon which this report is based may be found in Battelle Memorial Institute Laboratory Record Books Nos. 18749 and 19782.

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APPENDIX I

MELTING DATA

TABLE 40. MELTING DATA FOR ALLOY COMPOSITIONS SELECTED FOR EVALUATION

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Cast Hardness(a), VHN | Ingot | | Electrode Weight Change, grams | Number of Melts | Comments |
|---|-------------------|-----------------------------|------------------------------|---------------------------|---|-----------------------|-------------------|
| | | | Original Weight, grams | Final Weight, grams | | | |
| Dispersion Effectiveness | | | | | | | |
| 5W-2, 5Mo | 268B | 256 | 150,000 | 150,293 | -0.40 | 17 | Tungsten pickup |
| 5W-2, 5Mo | 268G | 213 | 150,001 | 149,855 | +0.03 | 8 | Sub for 268B |
| 5W-2, 5Mo-0, 07C | 311 | 274 | 150,001 | 150,312 | -0.27 | 11 | Tungsten pickup |
| 5W-2, 5Mo-0, 07C | 311A | 272 | 150,000 | 150,002 | -0.07 | 7 | Sub for 311 |
| 5W-2, 5Mo-0, 13C | 312 | 314 | 150,001 | 150,252 | -0.16 | 9 | Small shrink hole |
| 5W-2, 5Mo-0, 13C | 312A | 309 | 150,002 | 150,201 | -0.26 | 7 | (b) |
| 5W-2, 5Mo-1Hf | 319 | 228 | 150,001 | 149,876 | -0.02 | 7 | |
| 5W-2, 5Mo-2Hf | 320 | 242 | 150,001 | 149,890 | -0.03 | 7 | |
| 5W-2, 5Mo-0, 5Zr | 313 | 222 | 150,000 | 149,897 | 0.00 | 11 | |
| 5W-2, 5Mo-1Zr | 314 | 233 | 150,000 | 149,779 | 0.00 | 11 | |
| 5W-2, 5Mo-1Hf-0, 07C | 321 | 289 | 150,001 | 150,095 | -0.16 | 7 | |
| 5W-2, 5Mo-0, 5Zr-0, 07C | 315 | 294 | 150,002 | 150,336 | -0.26 | 13 | Tungsten pickup |
| 5W-2, 5Mo-0, 5Zr-0, 07C | 315I | 289 | 150,001 | 150,002 | -0.08 | 7 | Sub for 315 |
| 5W-2, 5Mo-0, 5Zr-0, 07C | 315A | 300 | 150,002 | 150,234 | -0.19 | 10 | Tungsten pickup |
| 5W-2, 5Mo-0, 5Zr-0, 07C | 315J | 283 | 150,002 | 150,098 | -0.09 | 7 | Sub for 315A |
| 5W-2, 5Mo-0, 5Zr-0, 13C | 318 | 333 | 150,001 | 150,042 | -0.05 | 7 | |
| 5W-2, 5Mo-1Zr-0, 07C | 317 | 373 | 150,001 | 150,720 | -0.95 | 7 | Tungsten pickup |
| 5W-2, 5Mo-1Zr-0, 07C | 317A | 299 | 150,002 | 149,921 | -0.01 | 7 | Sub for 317 |
| 5W-2, 5Mo-1Zr-0, 13C | 316 | 348 | 150,001 | 149,919 | -0.14 | 8 | |
| 5W-2, 5Mo-1Zr-0, 13C | 316A | 357 | 150,002 | 150,040 | -0.20 | 7 | |
| Repeat Dispersion Effectiveness | | | | | | | |
| 5W-2, 5Mo-1Hf-0, 07C | 321A | 276 | 150,000 | 149,771 | -0.01 | 8 | |
| 5W-2, 5Mo-1Hf-0, 07C | 321B | 276 | 150,000 | 150,145 | -0.31(c) | 9 | |
| 5W-2, 5Mo-0, 5Zr-0, 07C | 315L | 281 | 150,001 | 149,811 | -0.04 | 8 | |
| 5W-2, 5Mo-0, 5Zr-0, 07C | 315M | 276 | 150,000 | 149,889 | -0.04 | 9 | |
| 5W-2, 5Mo-0, 5Zr-0, 07C | 315N | 276 | 150,002 | 149,937 | -0.03 | 8 | |
| 5W-2, 5Mo-0, 5Zr-0, 07C | 315O | 279 | 150,001 | 150,035 | -0.07 | 8 | |
| 5W-2, 5Mo-0, 5Zr-0, 07C | 315P | 283 | 150,002 | 149,936 | -0.05 | 8 | |
| 5W-2, 5Mo-0, 5Zr-0, 07C | 315Q | 274 | 150,001 | 149,844 | -0.02 | 8 | |
| 5W-2, 5Mo-0, 5Zr-0, 13C | 318A | 314 | 150,001 | 150,065 | -0.09 | 8 | |
| 5W-2, 5Mo-1Zr-0, 07C | 317B | 289 | 150,000 | 149,852 | -0.02 | 8 | |
| 5W-2, 5Mo-1Zr-0, 07C | 317C | 285 | 150,000 | 149,922 | -0.07 | 8 | |
| 5W-2, 5Mo-1Zr-0, 13C | 316B | 325 | 150,002 | 149,906 | -0.05 | 8 | |
| Fabrication Variables | | | | | | | |
| 5W-2, 5Mo-0, 5Zr-0, 07C | 315B | 292 | 150,001 | 149,996 | -0.10 | 7 | |
| 5W-2, 5Mo-0, 5Zr-0, 07C | 315C | 297 | 150,000 | 150,014 | -0.08 | 7 | |
| 5W-2, 5Mo-0, 5Zr-0, 07C | 315D | 297 | 150,003 | 150,052 | -0.11 | 7 | |
| 5W-2, 5Mo-0, 5Zr-0, 07C | 315E | 291 | 150,002 | 149,991 | -0.07 | 10 | |

TABLE 40. (Continued)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Cast Hardness(a), VHN | Ingot | | Electrode Weight Change, grams | Number of Melts | Comments |
|---|-------------------|-----------------------------|------------------------------|---------------------------|---|-----------------------|--------------------|
| | | | Original Weight, grams | Final Weight, grams | | | |
| Fabrication Variables (Continued) | | | | | | | |
| 5W-2.5Mo-0.5Zr-0.07C | 315F | 295 | 150.000 | 149.963 | -0.01 | 9 | |
| 5W-2.5Mo-0.5Zr-0.07C | 315G | 317 | 150.002 | 150.372 | -0.50 | 7 | Tungsten pickup |
| 5W-2.5Mo-0.5Zr-0.07C | 315K | 283 | 150.001 | 149.924 | -0.03 | 7 | |
| 5W-2.5Mo-0.5Zr-0.07C | 315H | 292 | 150.001 | 149.172 | 0.00 | 7 | Sub for 315G |
| Transition Behavior | | | | | | | |
| 2.5Mo | 322 | 157 | 150.001 | 149.870 | -0.02 | 9 | |
| 5Mo | 99E | 217 | 150.001 | 149.876 | 0.00 | 9 | |
| 7.5Mo | 161D | 262 | 150.001 | 149.836 | 0.00 | 11 | |
| 10Mo | 100E | 308 | 150.000 | 149.927 | 0.00 | 9 | |
| 10W | 88D | 219 | 150.001 | 149.944 | -0.03 | 10 | |
| 15W | 165C | 261 | 150.000 | 149.713 | 0.00 | 11 | |
| 20W | 89D | 358 | 150.002 | -- | -0.06 | 10 | Large shrink hole |
| 20W | 89G | 304 | 150.000 | 149.680 | +0.02 | 7 | |
| 5.2W-2.7Mo | 323 | 225 | 150.001 | 149.877 | 0.00 | 9 | |
| 7.9W-4.1Mo | 324 | 280 | 150.000 | 149.903 | 0.00 | 10 | |
| 10.6W-5.6Mo | 325 | 315 | 150.002 | 149.863 | -0.02 | 9 | |
| Stress-Rupture Evaluation | | | | | | | |
| 100Ta | 191 | 88 | 150.000 | 149.928 | -0.02 | 7 | |
| 2.5Mo | 322A | 149 | 150.000 | 149.776 | 0.00 | 7 | |
| 2.5Mo | 322B | 148 | 150.000 | 149.816 | +0.01 | 7 | |
| 5Mo | 99F | 218 | 150.001 | 149.857 | 0.00 | 7 | |
| 5Mo | 99G | 215 | 150.001 | 149.834 | +0.04 | 7 | |
| 7.5Mo | 161E | 253 | 150.001 | 149.612 | +0.01 | 10 | |
| 10Mo | 100F | 302 | 150.002 | 149.983 | 0.00 | 7 | |
| 5W | 164C | 155 | 150.000 | 149.900 | +0.01 | 7 | |
| 10W | 88E | 210 | 150.001 | 149.857 | +0.03 | 7 | |
| 20W | 89E | 312 | 150.001 | 149.507 | +0.01 | 7 | |
| 20W | 89F | 299 | 150.000 | 149.756 | +0.01 | 7 | |
| 5W-2.5Mo | 268C | 216 | 150.001 | 149.935 | 0.00 | 7 | |
| 5W-5Mo | 227C | 260 | 150.001 | 149.866 | +0.01 | 7 | |
| 5W-5Mo | 227D | 268 | 150.001 | 149.904 | 0.00 | 7 | |
| 5W-7.5Mo | 331 | 315 | 150.001 | 149.915 | 0.00 | 7 | |
| 5W-7.5Mo | 331A | 313 | 150.001 | 149.862 | +0.01 | 7 | |
| 10W-2.5Mo | 262C | 278 | 150.000 | 149.918 | 0.00 | 7 | |
| 10W-2.5Mo | 262D | 273 | 150.001 | 149.866 | 0.00 | 7 | |
| 10W-5Mo | 179F | 308 | 150.001 | 149.878 | 0.00 | 7 | Broke on machining |
| 10W-5Mo | 179G | 306 | 150.002 | 149.839 | 0.00 | 7 | |
| 15W-2.5Mo | 326 | 320 | 150.001 | 149.861 | 0.00 | 11 | |
| 15W-2.5Mo | 326A | 350 | 150.001 | 149.990 | -0.18 | 7 | |

TABLE 40. (Continued)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Cast Hardness ^(a) , VHN | Ingot | | Electrode | Number of Melts | Comments |
|---|-------------------|--|------------------------------|---------------------------|----------------------------|-----------------------|---------------------|
| | | | Original Weight, grams | Final Weight, grams | Weight Change, grams | | |
| <u>Welding Variables</u> | | | | | | | |
| 5Mo | 99H | 215 | 150,000 | 149,900 | -0.02 | 7 | Fabrication failure |
| 5Mo | 99I | 215 | 150,000 | 149,859 | -0.01 | 7 | |
| 5Mo | 99J | 218 | 150,001 | 149,900 | 0.00 | 7 | |
| 12,5W | 266B | 242 | 150,000 | 149,839 | 0.00 | 7 | |
| 12,5W | 266C | 242 | 150,001 | 149,765 | +0.01 | 7 | |
| 12,5W | 266D | 240 | 150,000 | 149,833 | -0.02 | 7 | |
| 5W-2,5Mo | 268D | 221 | 150,001 | 149,887 | 0.00 | 7 | |
| 5W-2,5Mo | 268E | 222 | 150,000 | 149,878 | -0.01 | 9 | |
| 5W-2,5Mo | 268F | 224 | 150,001 | 149,868 | 0.00 | 7 | |
| 5W-2,5Mo | 268H | 210 | 150,001 | 149,874 | 0.00 | 7 | |
| 10W-2,5Mo | 262E | 266 | 150,001 | 149,752 | 0.00 | 7 | |
| 10W-2,5Mo | 262F | 270 | 150,001 | 149,867 | -0.01 | 7 | |
| 10W-2,5Mo | 262G | 258 | 150,001 | 149,895 | 0.00 | 7 | |
| <u>Screening Alloys</u> | | | | | | | |
| 5W-2,5Ru | 329 | 280 | 149,973 | 149,758 | 0.00 | 9 | |
| 5W-5Re | 328 | 304 | 150,001 | 149,860 | 0.00 | 9 | |
| 5W-2,5Mo-2,5Re | 330 | 278 | 150,001 | 149,801 | 0.00 | 9 | |
| 7,5W-2,5Re | 327 | 254 | 150,000 | 149,787 | 0.00 | 11 | |

(a) Hardness values are the average of five impressions using a 10-kg load.

(b) Tungsten pickup. Substitute for 312.

(c) Electrode loss high; however, hardness value indicates good compositional control.

TABLE 41. CHEMICAL ANALYSES OF TANTALUM-BASE ALLOYS (a)

| Nominal Composition (Balance Tantalum), weight per cent | Alloy Specimen | Amount Present, weight per cent | | | | | |
|---|-----------------------|---------------------------------|--------|-------|-----------------------|------|------|
| | | C | O | N | H | W | |
| 5W-2.5Mo | 268G | 0.011 | 0.0003 | 0.003 | 0.0001 | -- | -- |
| 5W-2.5Mo-0.07C | 311A | 0.08 | 0.0005 | -- | 0.0001 | -- | -- |
| 5W-2.5Mo-0.13C | 312A | 0.16 | 0.0021 | 0.002 | 0.0009 | -- | -- |
| 5W-2.5Mo-1Hf | 319 | -- | 0.0013 | 0.005 | 0.0002 | -- | 1.02 |
| 5W-2.5Mo-2Hf | 320 | -- | 0.0009 | 0.005 | 0.0001 | -- | 2.00 |
| 5W-2.5Mo-0.5Zr | 313 | -- | 0.0004 | 0.004 | 0.0001 | -- | 0.55 |
| 5W-2.5Mo-1Zr | 314 | -- | 0.0013 | 0.003 | 0.0002 | -- | 0.97 |
| 5W-2.5Mo-1Hf-0.07C | 321 | 0.09 | 0.0028 | 0.003 | 0.0006 | -- | 2.40 |
| 5W-2.5Mo-0.5Zr-0.07C | 315B-H ^(b) | 0.08 | 0.0021 | 0.001 | 0.0005 | 4.90 | 0.52 |
| 5W-2.5Mo-0.5Zr-0.07C | 315C-H | 0.08 | 0.0010 | 0.002 | 0.0006 | 4.82 | 0.56 |
| 5W-2.5Mo-0.5Zr-0.07C | 315D-H | 0.09 | 0.0017 | 0.005 | 0.0007 | 4.92 | 0.55 |
| 5W-2.5Mo-0.5Zr-0.07C | 315E-H | 0.09 | 0.0016 | 0.002 | 0.0007 | 4.92 | 0.52 |
| 5W-2.5Mo-0.5Zr-0.07C | 315F-H | 0.09 | 0.0021 | 0.003 | 0.0006 | 4.87 | 0.50 |
| 5W-2.5Mo-0.5Zr-0.07C | 315H-H | 0.08 | 0.0022 | 0.003 | 0.0007 | 4.98 | 0.53 |
| 5W-2.5Mo-0.5Zr-0.07C | 315K | 0.07 | 0.0010 | 0.003 | 0.0025 ^(c) | 4.92 | 0.56 |

(a) Analyses conducted on as final-rolled sheet.

(b) Additional letter identification indicates "H" for 980 C (1800 F) final rolling.

(c) High hydrogen content probably picked up from 1650 C (3000 F) fabrication. Vacuum annealing should lower hydrogen content to <0.0010 per cent prior to testing.

APPENDIX II

WELDING STUDIESTABLE 42. AUTOMATIC TIG WELDING CONDITIONS FOR DISPERSION EFFECTIVENESS ALLOYS^(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Arc Voltage, volts | Arc Current, amperes |
|---|-------------------|-----------------------|-------------------------|
| 5W-2.5Mo | 268G | 16 | 76 |
| 5W-2.5Mo-1Hf-0.07C | 321A | 17 | 78 |
| 5W-2.5Mo-0.5Zr-0.07C | 315N | 16 | 82 |
| 5W-2.5Mo-0.5Zr-0.13C | 318A | 16.5 | 83 |
| 5W-2.5Mo-1Zr-0.07C | 317B | 16 | 82 |
| 5W-2.5Mo-1Zr-0.13C | 316B | 16 | 96 |

^(a) Helium atmosphere; 7-ipm travel speed.

TABLE 43. AUTOMATIC TIG WELDING CONDITIONS FOR TANTALUM-BASE ALLOYS SELECTED FOR DETAILED EVALUATION^(a)

| Alloy Composition (Balance Tantalum), weight per cent | Alloy Specimen | Arc Voltage, volts | Arc Current, amperes |
|---|-------------------|-----------------------|-------------------------|
| 5Mo | 99H | 16.5 | 80 |
| | 99I-1 | 15 | 78 |
| | 99I-2 | 18 | 80 |
| | 99J-1 | 18 | 80 |
| | 99J-2 | 18 | 80 |
| 12.5W | 266B | 18 | 88 |
| | 266C-1 | 15 | 86 |
| | 266C-2 | 15 | 87 |
| | 266D-1 | 16 | 88 |
| | 266D-2 | 19 | 85 |
| 5W-2.5Mo | 268D(b) | 13, 13.5 | 93, 110 |
| | 268D(c) | 17 | 98 |
| | 268E-1 | 15.5 | 94 |
| | 268E-2 | 15 | 94 |
| | 268H-1 | 16 | 95 |
| | 268H-2(d) | 17 | 90 |
| | 268H-2(c) | 15 | 94 |
| 10W-2.5Mo | 262E | 17 | 100 |
| | 262F | 17 | 94 |
| | 262G(d) | 15 | 90 |
| | 262G(c) | 15 | 98 |

(a) Helium atmosphere; 7-ipm travel speed.

(b) First pass in argon one-half of strip at 13 volts and 93 amperes; other half of strip at 13.5 volts and 110 amperes.

(c) Second pass

(d) First pass.

APPENDIX III

RECRYSTALLIZATION BEHAVIOR

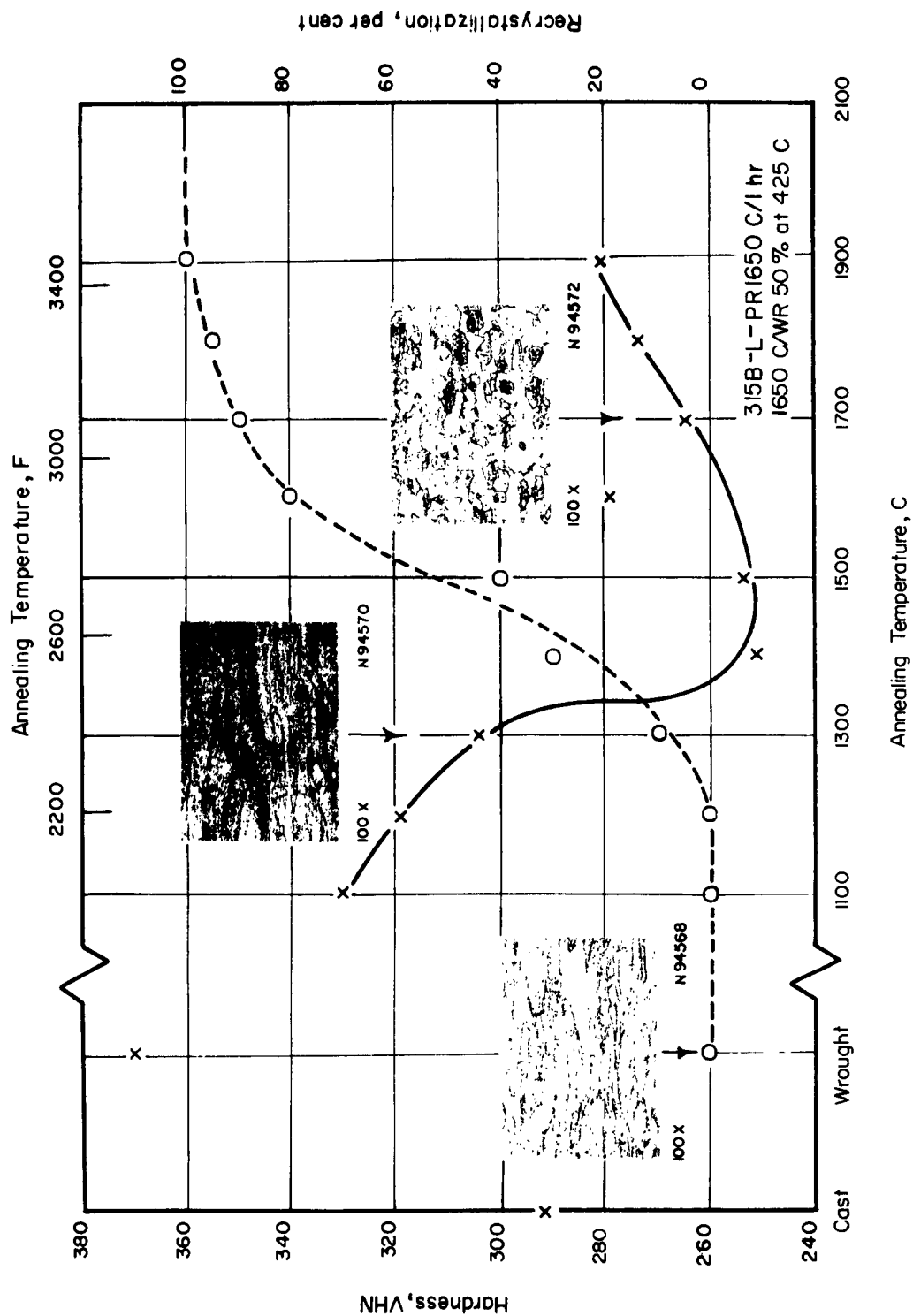


FIGURE 30. EFFECT OF ANNEALING TEMPERATURE ON THE RECRYSTALLIZATION BEHAVIOR OF Ta-5W-2.5Mo-0.5Zr-0.07C

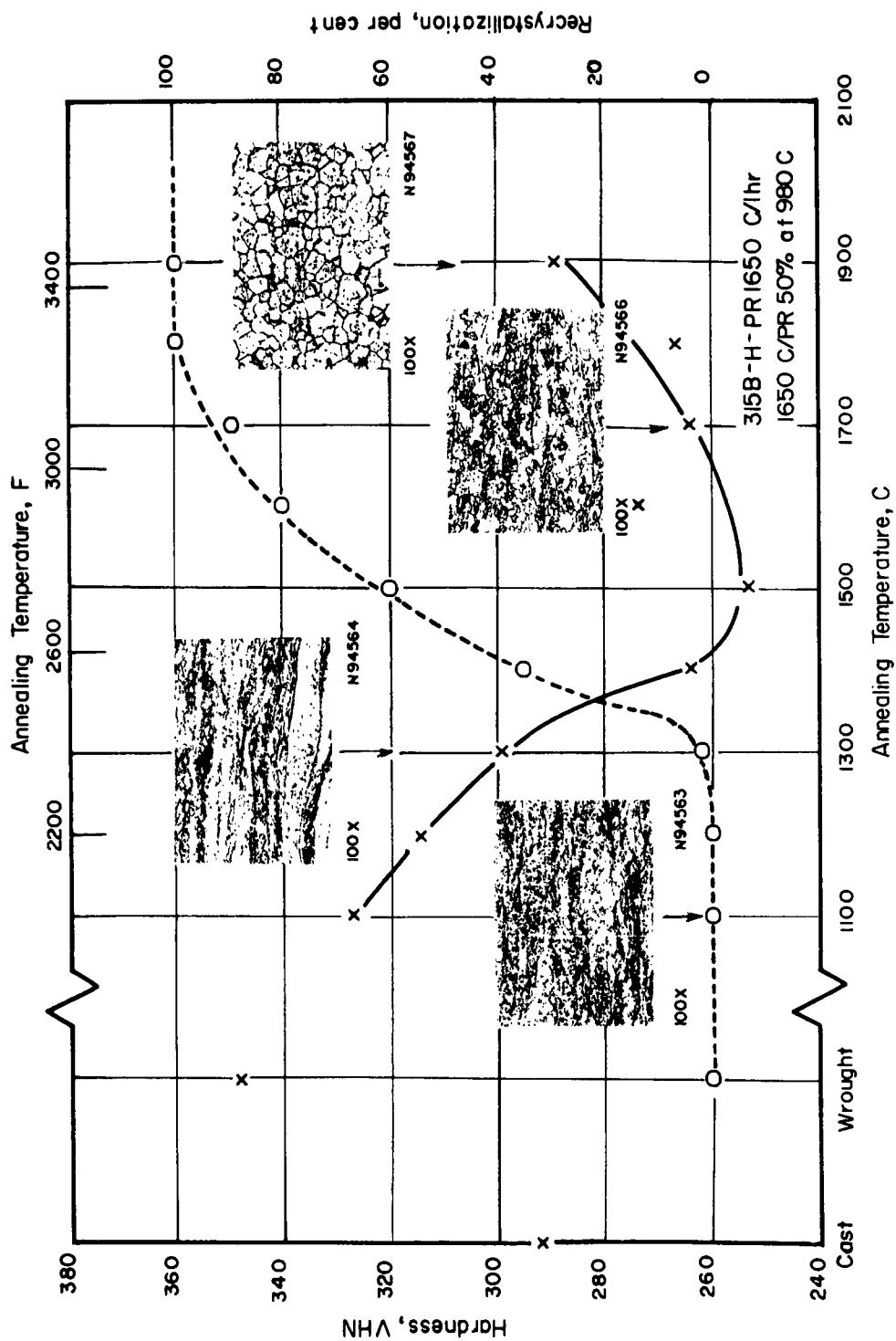


FIGURE 30. (CONTINUED)

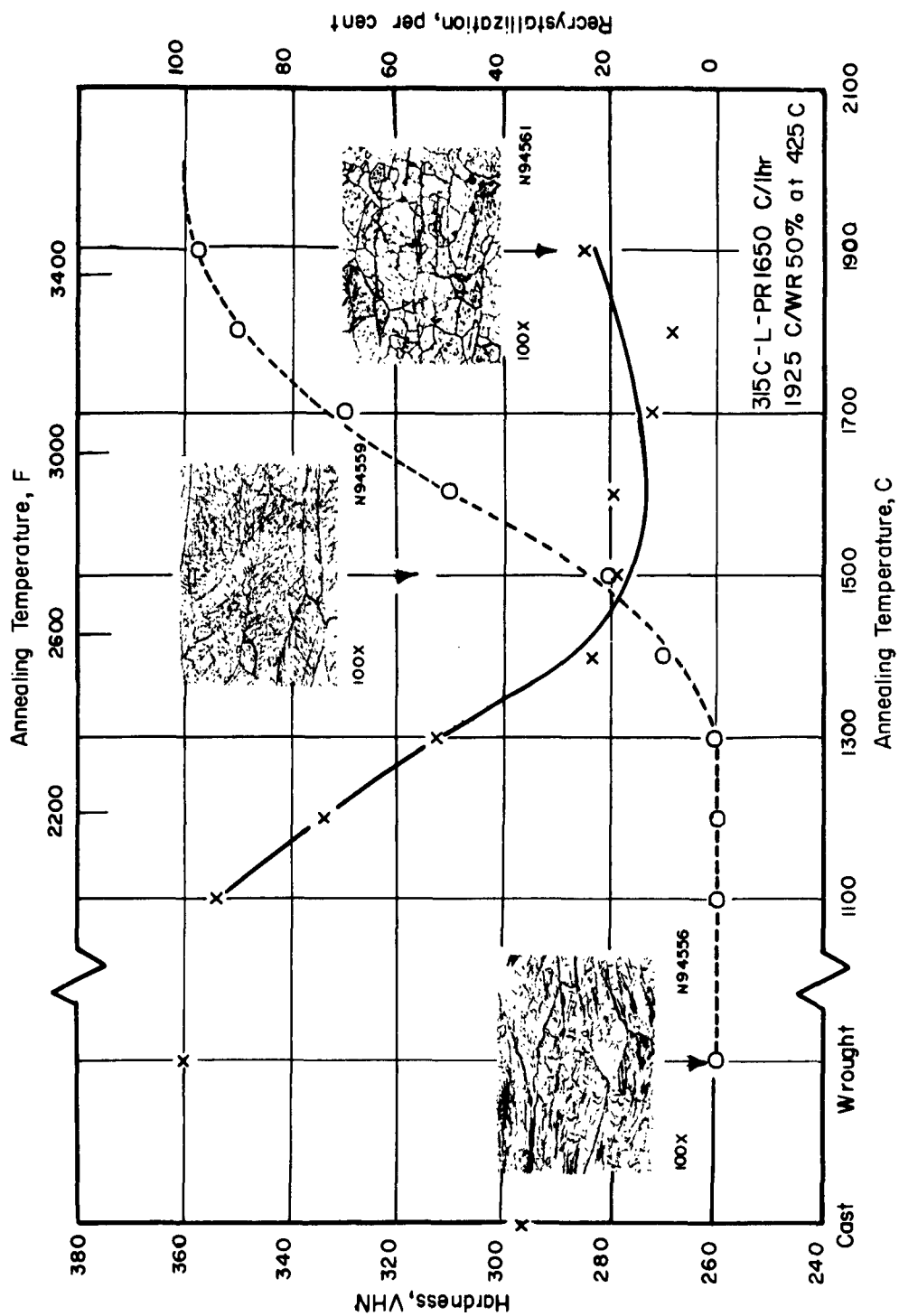


FIGURE 30. (CONTINUED)

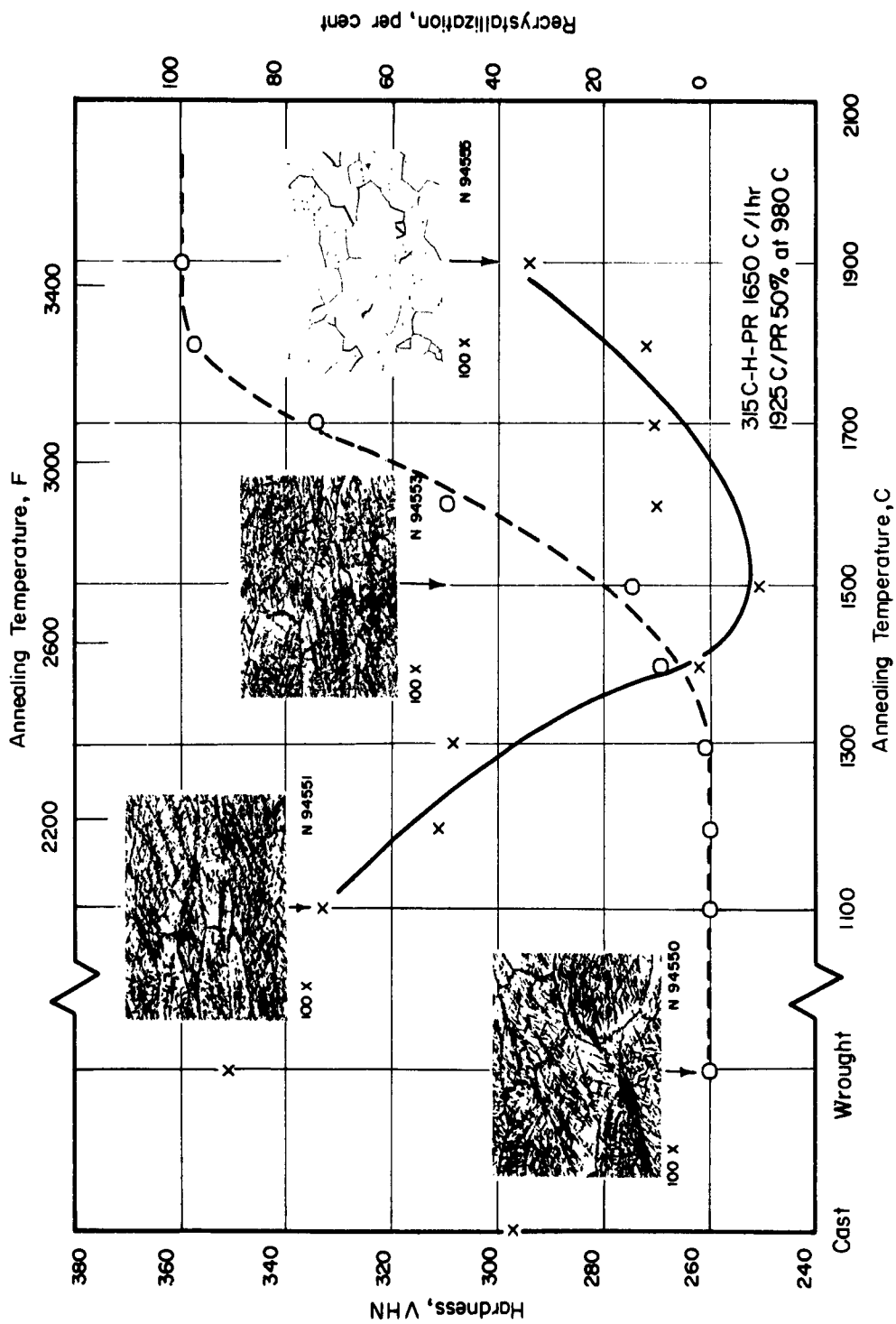


FIGURE 30. (CONTINUED)

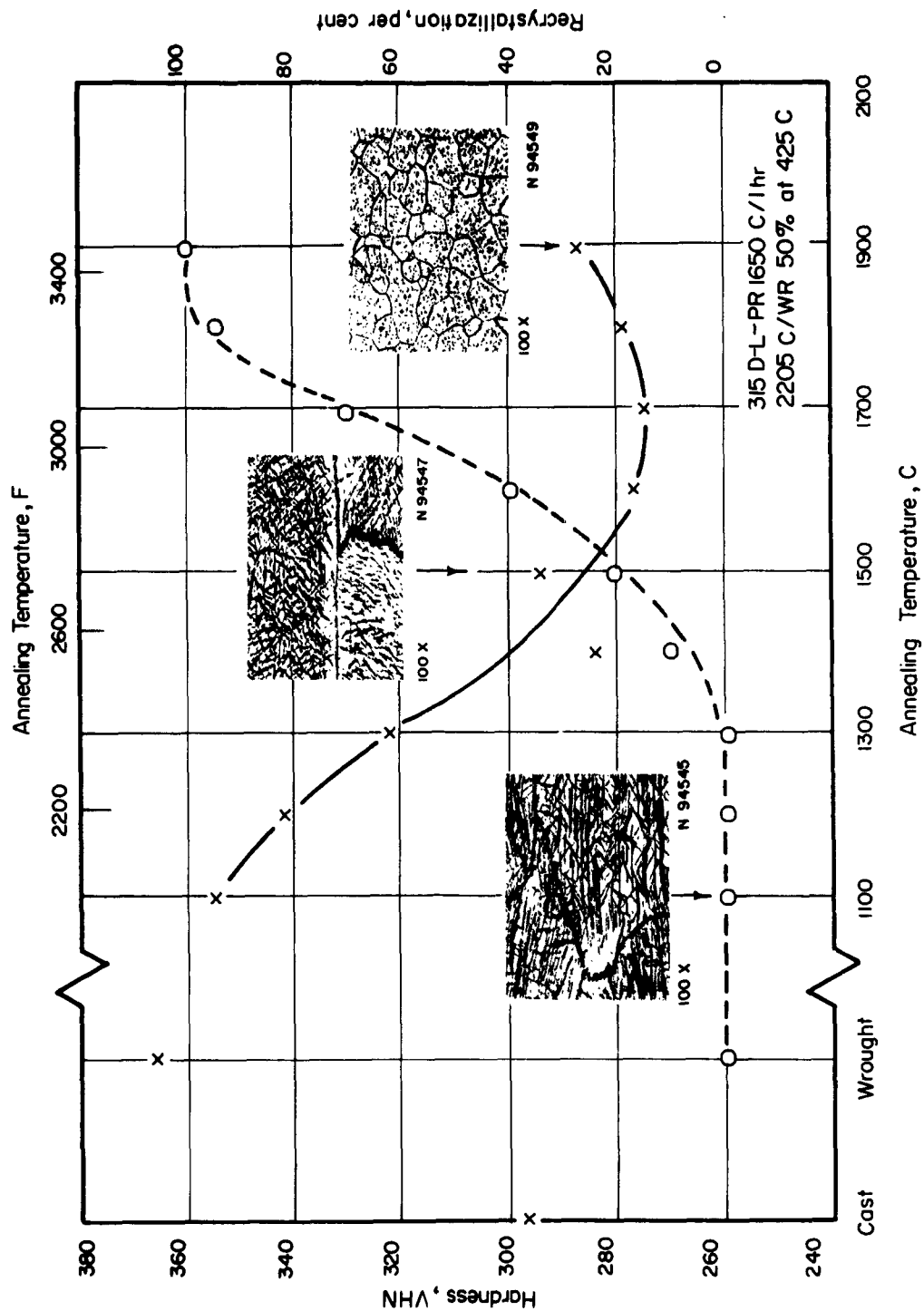


FIGURE 30. (CONTINUED)

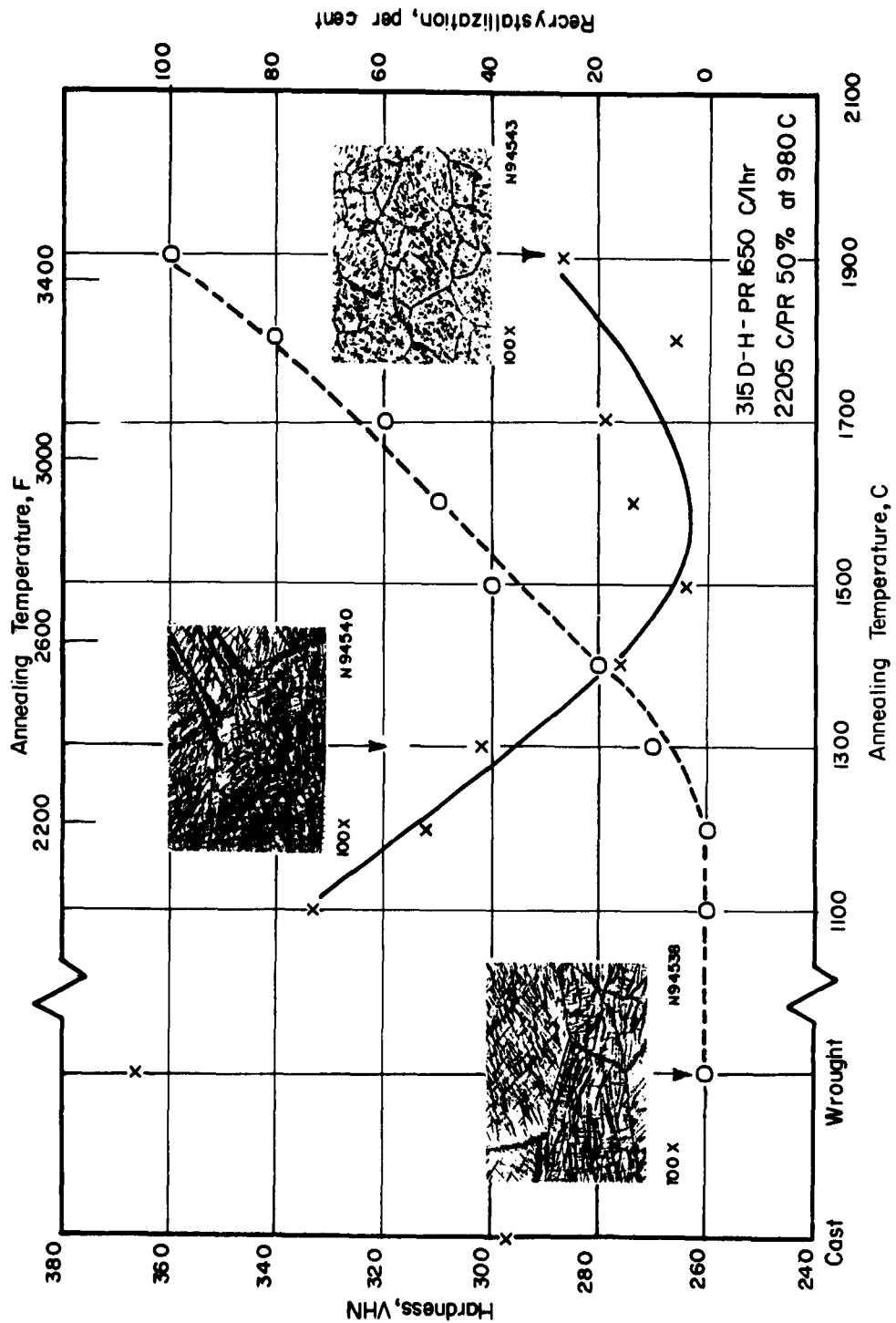


FIGURE 30. (CONTINUED)

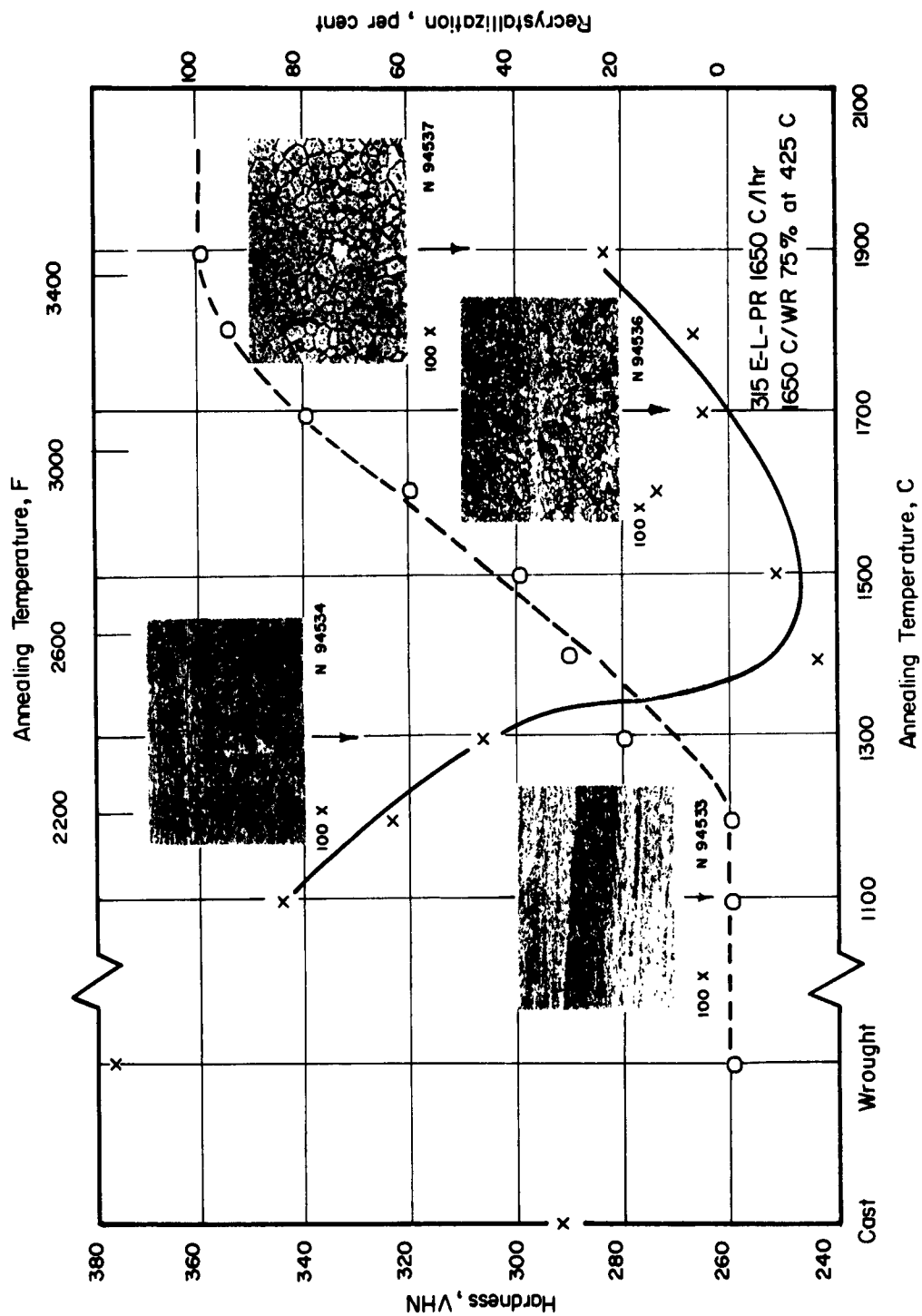


FIGURE 30. (CONTINUED)

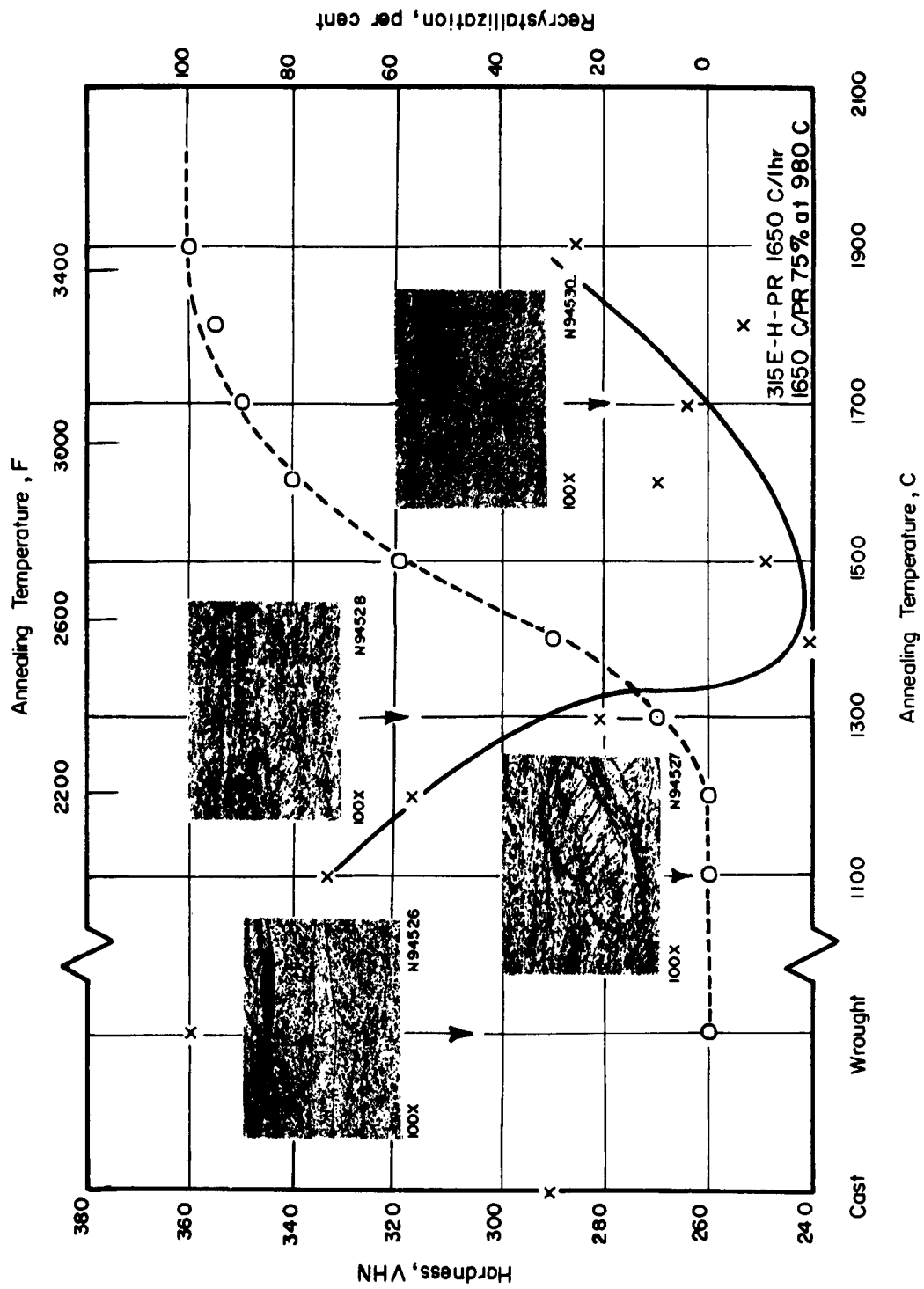


FIGURE 30. (CONTINUED)

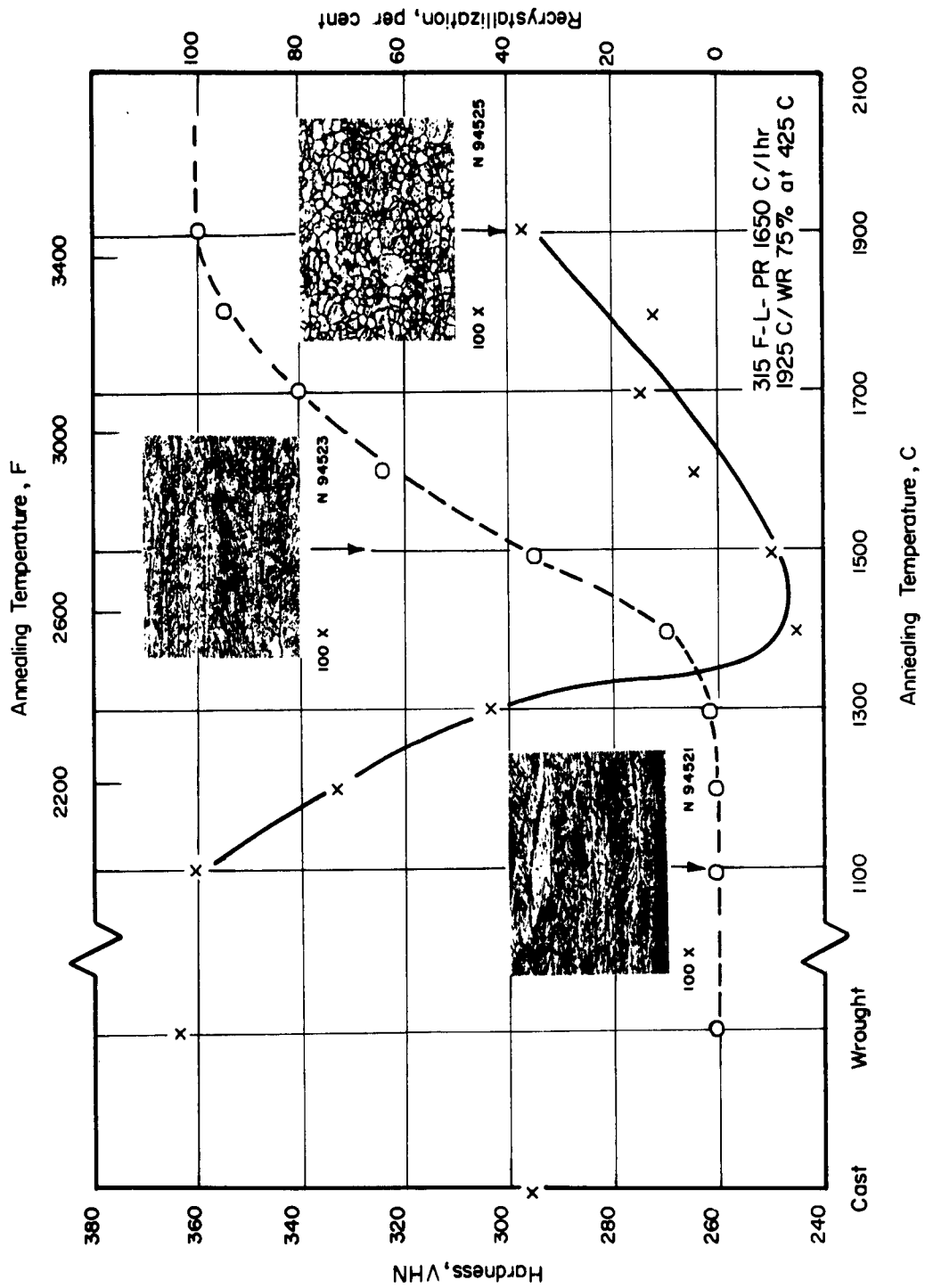


FIGURE 30. (CONTINUED)

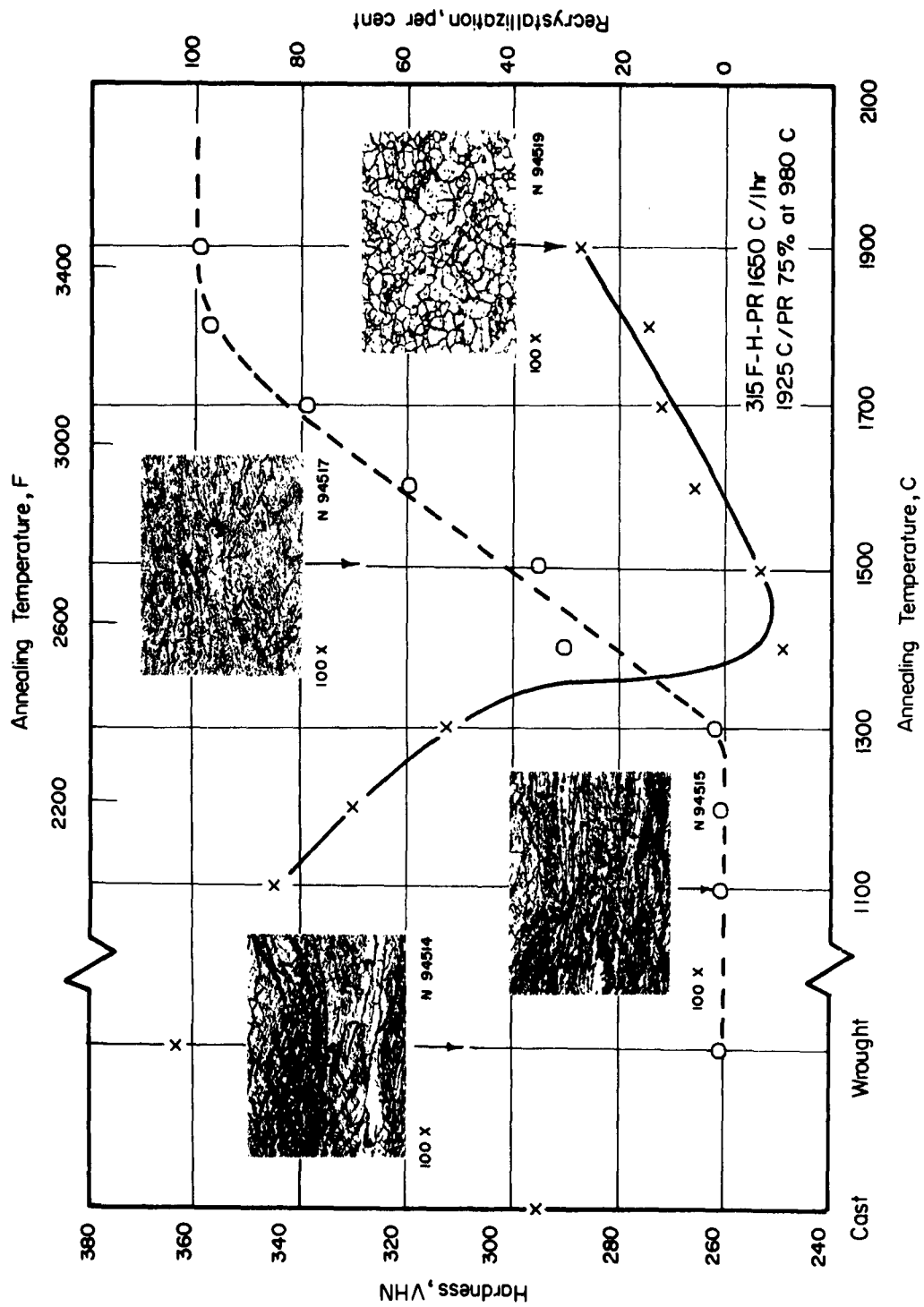


FIGURE 30. (CONTINUED)

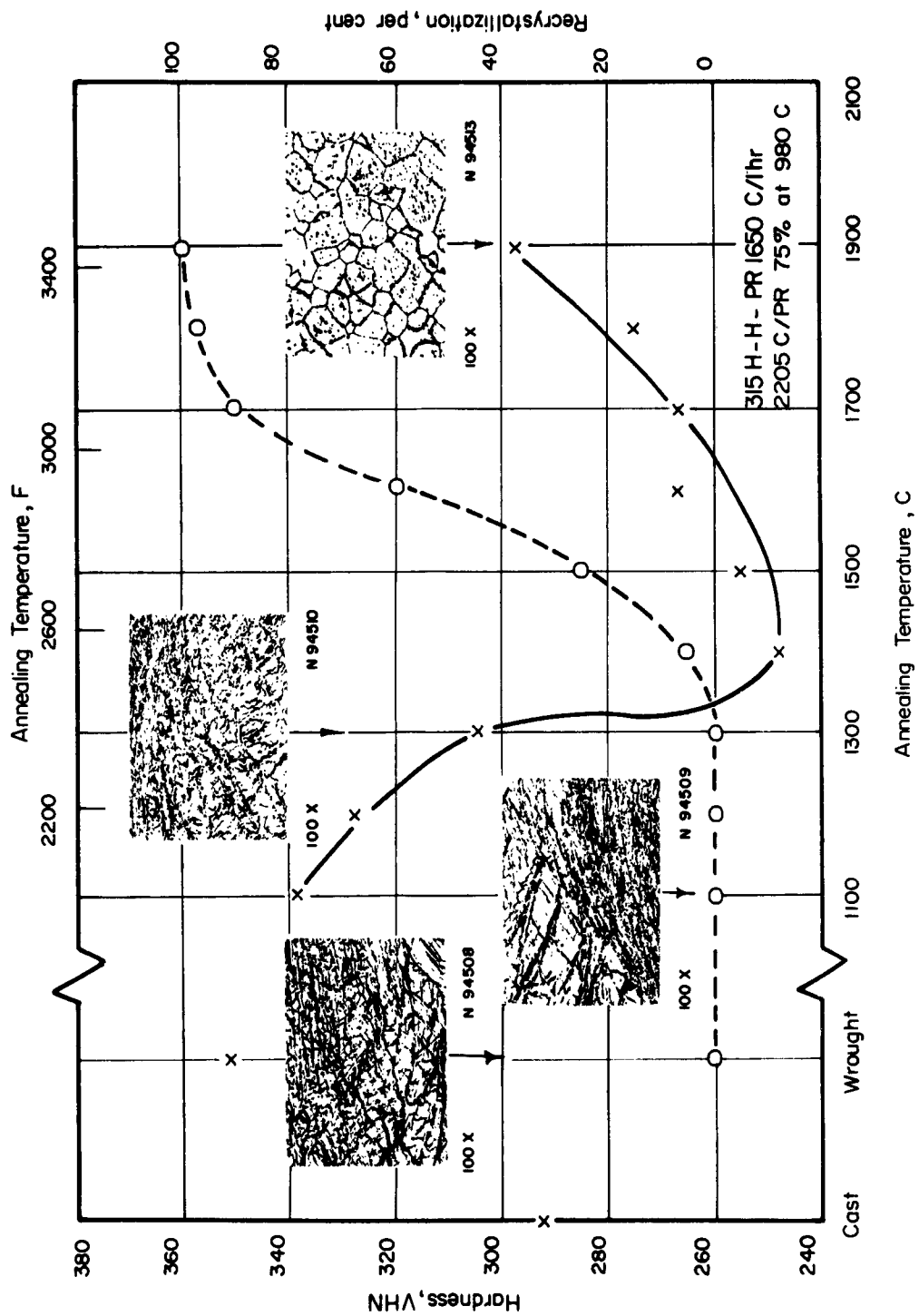


FIGURE 30. (CONTINUED)

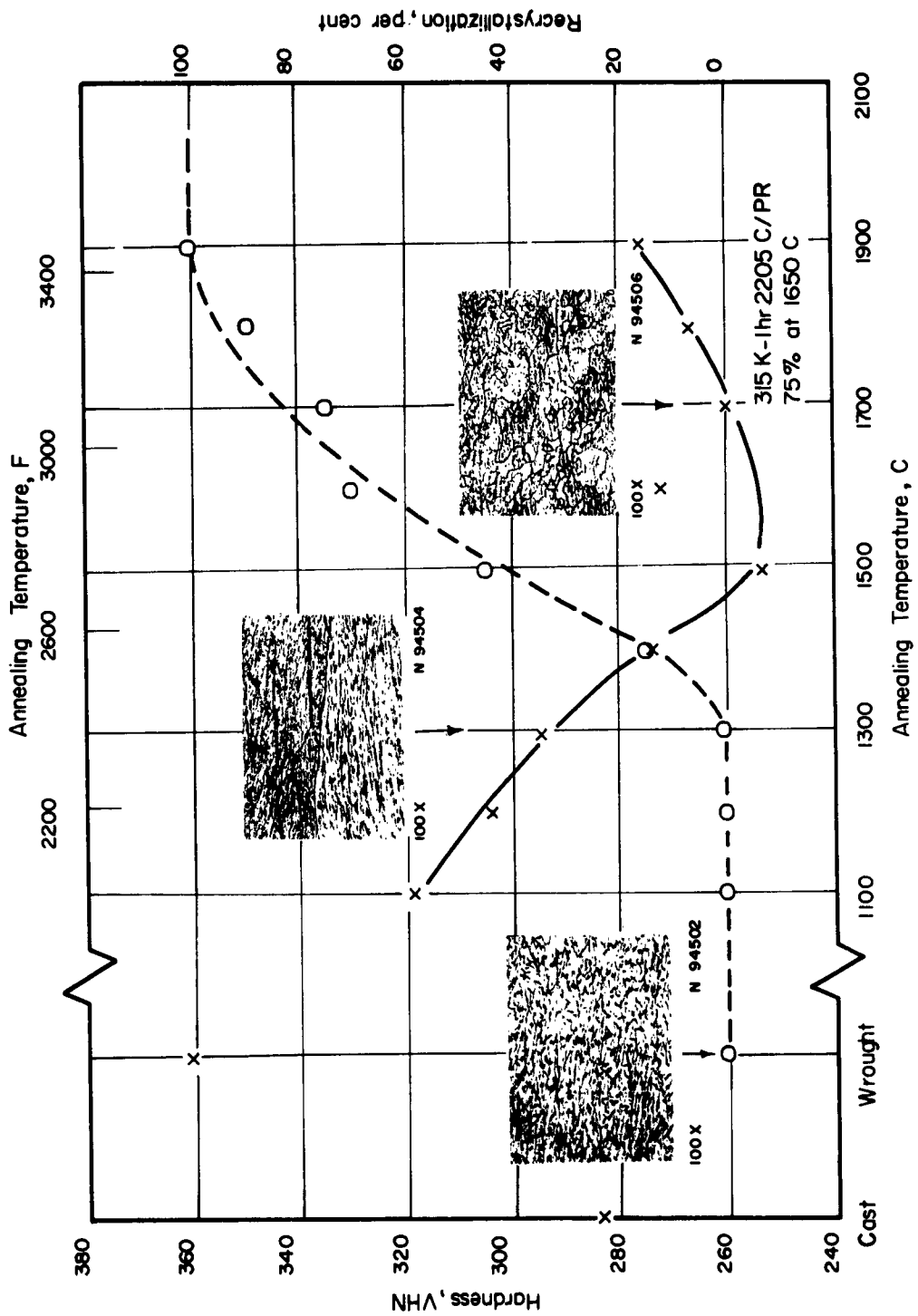


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| <p>atures (1480 C(2700F)). Solution-process anneals for alloys containing "ZrC dispersion" greatly increase strength at 1480 C (2700 F), but seriously impair fabricability and ductility. Molybdenum is a less effective strengthener than tungsten as an alloying addition to tantalum. Tungsten additions provide higher stress-rupture strengths with less degrading effects on low-temperature ductility than do equivalent atomic percentages of molybdenum. Welding increases the ductile-to-brittle transition temperature of Ta-W-Mo alloys by 300 to 500 C(540 to 900F). Rhenium and ruthenium additions showed little or no superiority to tungsten as solid-solution strengtheners, when both high- and low-temperature effects were considered.</p> | <ol style="list-style-type: none"> VI. In ASTIA collection | <p>atures (1480 C(2700F)). Solution-process anneals for alloys containing "ZrC dispersion" greatly increase strength at 1480 C (2700 F), but seriously impair fabricability and ductility. Molybdenum is a less effective strengthener than tungsten as an alloying addition to tantalum. Tungsten additions provide higher stress-rupture strengths with less degrading effects on low-temperature ductility than do equivalent atomic percentages of molybdenum. Welding increases the ductile-to-brittle transition temperature of Ta-W-Mo alloys by 300 to 500 C(540 to 900F). Rhenium and ruthenium additions showed little or no superiority to tungsten as solid-solution strengtheners, when both high- and low-temperature effects were considered.</p> | <ol style="list-style-type: none"> VI. In ASTIA collection |